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(NASA-CR-162054) SPACE ENVIRONMENTAL  
EFFECTS: CONSTRUCTION AND UTILIZATION OF A  
SYSTEM TO MEASURE LOW THERMAL STRAIN IN ONE  
METER GRAPHITE EPOXY TUBES Final Report,  
Nov. 1979 - Nov. 1981 (Alabama Univ.,

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**FINAL REPORT**

**November 1979 - November 1981**

**Contract NAS8-33377**

**SPACE ENVIRONMENTAL EFFECTS**

**Construction and Utilization of a System  
to Measure Low Thermal Strain in  
One Meter Graphite Epoxy Tubes**

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## ABSTRACT

A system for measuring the expansion of low coefficient of thermal expansion (CTE) materials has been constructed around a H.P. 5526-A laser measuring system. The vacuum ( $\sim 1\mu$ ) CTE measurements in the  $-150^{\circ}\text{F}$  to  $+120^{\circ}\text{F}$  range were made over a 6 month period on a 2.3" O.D. x 1/16" wall by  $\sim .9$  m long, 59° wrap, graphite epoxy (G/E) tube yielding CTE ( $\alpha$ ) values of  $2.5$  to  $5 \times 10^{-7}/^{\circ}\text{F}$  above ambient and  $2 \pm 1 \times 10^{-7}/^{\circ}\text{F}$  below ambient temperature. To assure that the below ambient,  $\sim 10\mu$  high open loop nature of the  $\Delta L/L$  vs.  $T$  curves was not apparatus related, similar size quartz tubes (A and B) were checked and found to have only a  $2\mu$  (negligable for quartz) open loop component. These two quartz tubes, A and B, had ambient CTE values 20% and 45% respectively higher than the average handbook value ( $.305 \times 10^{-6}/^{\circ}\text{F}$ ). The overnight microcreep diminished an order of magnitude during the first several cycles after the system had been reopened.

#### ACKNOWLEDGMENTS

The conception, design, construction and pretesting of the low CTE measuring system was done by Graduate Research Assistants Mr. L. Dickerson and Mr. Don A. Gregory with guidance from Prof. J. G. Castle. As the coefficient of thermal expansion (CTE) system evolved into the operational phase, Graduate Research Assistant Ms. Carol J. Rives, with the help of Ms. Kathy Bogush and Mr. Mark Gordon, performed CTE measurements while refining the system. Mr. Bobby Cothren provided expert technical assistance while Mrs. A. F. Whitaker's scientific and administrative aid was appreciated.

#### BACKGROUND

NASA requires ultra high dimensional stability for the Space Telescope, especially in the space orbital environment's wide temperature extremes. One candidate material, Graphite fiber (negative CTE) filled epoxy, is clad at an angle of about  $59^\circ$  to just annul the expansion along the tube axis. Such a sample was received indirectly from Boeing as a verification strut for checking thermal expansion from  $-150^\circ\text{F}$  to  $120^\circ\text{F}$ .

**Sample Description**

**MATERIALS**

T-50 (PAN)/934 (FIBERITE HyE 2134)  
T-300/934 (FIBERITE HyE 1034C)

**LAMINATE DESCRIPTION**

$\pm 59^\circ$  T-300       $0^\circ$   $2_{T-50}$  S

**CURE CYCLE**

275°F/100 PSI (AUTOCLAVE) for 4 hours plus  
275°F (OVEN) for 8 hours

**TUBE SIZE**

2.3" I.D. x 1/16" wall STRUT DESIGNATION .002

**NUMBER ON TUBE**

MTS-DS 011-1-02 3

**SAMPLES**

**QUARTZ**

TUBE-"A"                      AMERSIL                      COMMERCIAL  
~ 60 mm x 2 mm wall x ~ 3' - from Stock on Hand Diamond saw cut.

TUBE-"B"                      AMERSIL                      COMMERCIAL  
TO8 ~ 60 mm O.D. x 2 mm wall x ~ 38" ordered for replacement  
Diamond saw cut on 1 end and fire polished on same end.

## EQUIPMENT

ITEM	MFG	MOD	SERIAL #
Laser Display	H.P.	5505A	(MSFC81923)1324A00580
Laser Head	H.P.	5500C	
TC Temperature Display	Digimite	Multimite	(NASA105796)
TC Pressure Gauge	Veeco	TG-70	00-5882-02210
Temperature Recorder	Leeds and Northrop Co	Speedomax with Recorder	(MSFC75170)D71-6891b-2-3
Vacuum Pump	Welch Scientific Co.	1397	12613 (No Trap)
Digital Recorder	H.P.	5050A	MSFC-
Vacuum Gauge	Veeco	DV-1m	
Resolution Extender	H.P.	K04-58815A	S-7881
Retroreflectors	H.P.	10556A(?)	
T.C. wire Cu Constantan		Size 28 gauge, Insulation	~(.04"x.06")



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### INTRODUCTION

A precise laser (Michelson interferometer) method of measuring small ( $\pm .0157\mu$  resolution) expansions of the sample is used. The two non-transmitting mirrors (Figure 3) are located by (folding the beam  $90^\circ$  into parallel with the other beam with an extra mirror) on each end of the sample ( $\frac{1}{2}$  the increase in optical path). The change in sample length is displayed (and printed) in units of  $\lambda/40$  ( $.0157\mu$ ). Other changes in the optical path, which would normally occur with humidity and atmospheric pressure changes, have been completely eliminated by enclosing the sample in a vacuum box. Such long term stability is needed to measure a microcreep of  $2 \mu$  over 24 hours.

### VACUUM BOX

The 1' x 1' x 3' quartz window vacuum box holds its vacuum with no signs of leaks at the  $1\mu$  range. However, after about ten minutes with the pump valve shut, the system pressure rises to about  $10\mu$ . A calibrated adjustable leak valve has been installed to vent in nitrogen, argon, or helium gas to smooth the thermal gradients. An end cap has been built to hold a 6" longer sample. A Cu base plate has been removed since it distorted with changing temperature causing erratic outputs. This plate was replaced by a granite slab having the required dimensional stability. See Figure 1.

In order to check microcreep and friction, one of the sample knife edges has been replaced by a quartz (for low thermal conductivity) cylinder roller. A second contoured roller has been machined from Corning "Machinable Glass" rod for better cradling the graphite/epoxy

tube while providing only one degree of frictionless freedom (longitudinal).

At the heart of the expansion measurements is a dual beam (two frequency) Hewlett Packard Model 5526A Laser Interferrometer system with accessories. This system functioned nicely and the retroreflectors relaxed the strict alignment requirements on the two end mirrors.

This system, especially the retroreflectors, was designed for near ambient operation hence these end retroreflectors were allowed only  $\sim 1/3$  the temperature excursion of the center of the sample. This produced a rather sharp, unwanted temperature gradient ( $< 10^\circ\text{F}/\text{cm}$ ) along the tube's outside few inches which may cause some error in  $\alpha(\text{CTE})$ ; however, this arrangement reduces the large errors due to greater expansion of mirror mounts and retroflectors connections, with the 3 x larger temperature excursions required for the isothermal system.

This long ( $\sim 9\text{m}$ ) sample capability provided an order of magnitude reduction in the percentage of error from end effects.

Four or five copper constantan 12.5 mil. thermocouples were mounted on the sample. The average of the 3 located at positions ( $1/4$ ,  $2/4$ ,  $3/4$ ) of the length of the sample were averaged to provide the sample temperature graphed in Figs. 4 and 6. One or two other thermocouple junctions were located near the sample in various places (depending on the run) such as on the brass tube, on the sample end, on the invar mirror (retroreflector) mount or on the granite slab.

In order to preserve the sample integrity, no holes were drilled in the sample for the thermocouple junctions but silicon thermal conducting

## SUPPORT SYSTEM

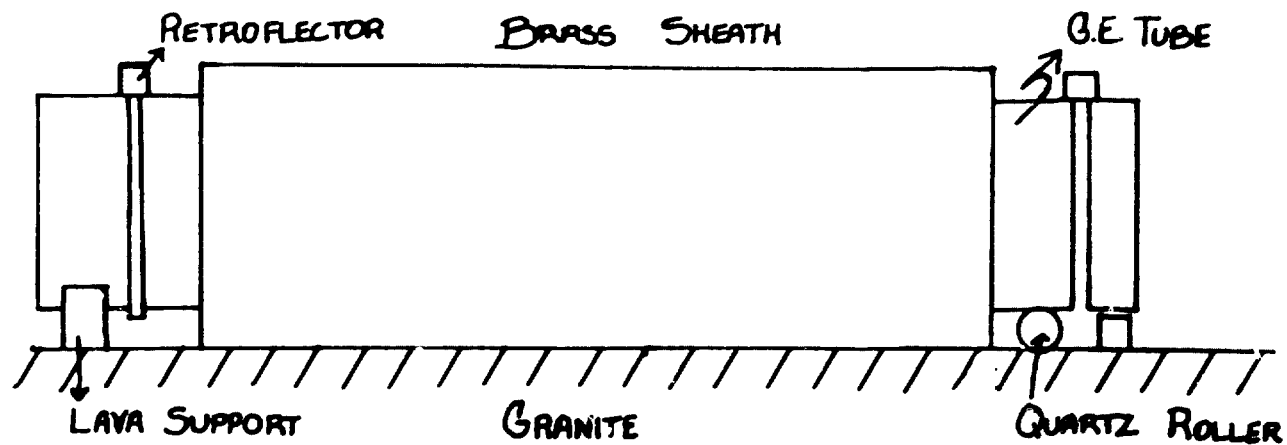
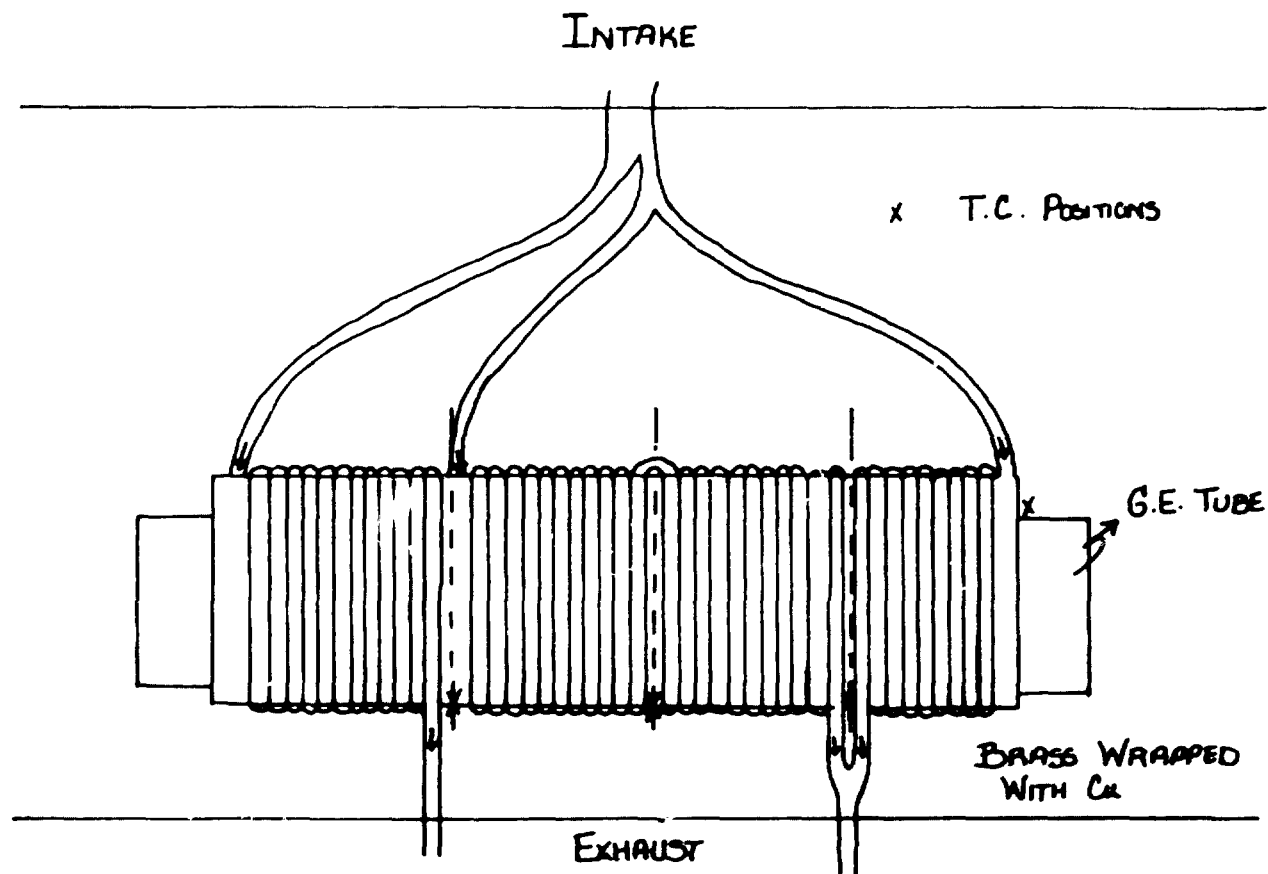


Figure 1. The sample was free except for two end supports and a few (4 or 5) thermocouple wires. The granite was needed for stability against previous thermal warping.

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Figure 2. Sample temperature control is provided by  $N_2$  gas drawn either from a  $LN_2$  tank or from heated coils. Exhaust valves (not shown) allowed for some regulation on one end of the sample.



HEATING/COOLING SYSTEM

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Figure 3. Two arms of a Michaelson Interferometer measure only relative displacement of the sample end mirrors (retroreflectors).

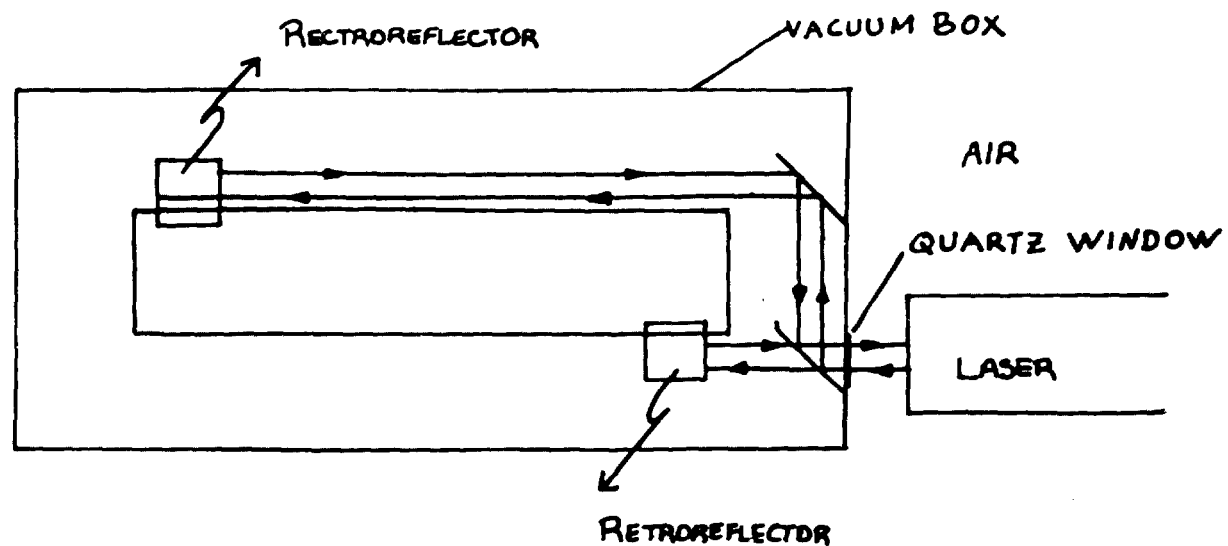


Figure III - Laser Path

paste was used along with a rubber band or a styrofoam cup to hold the thermocouple junction to the bottom of the sample tube.

In order to help insure quasithermal-equilibrium the serious data points were only taken as those with  $\dot{N} \leq .05 \text{ sec}^{-1}$ .  $\dot{N}$  is averaged over a 5 second period.  $\dot{N} < .05 \text{ sec}^{-1}$  implies  $\dot{L} < .047 \mu/\text{minute}$  or  $\dot{T} \leq 0.1 \text{ or } 0.2 \text{ }^\circ\text{F}/\text{minute}$ . Here  $N$ , the counter reading, is thought of as a unit of length equal to  $\lambda/40$  (.0157 $\mu$ ).

#### TEMPERATURE CONTROL OF SAMPLE

Sample heating and cooling is provided as seen in Figure 2 by air through 1/4" O.D. Cu pipe wound in a coaxial (with the sample) helix. Cool  $N_2$  is drawn from a liquid nitrogen tank while hot air is provided by wrapping part of the external 1/4" pipe line with a heating tape. Above ambient temperature (see Figs. 15 and 16) the sample has some temperature gradient while below, the sample temperature varies by about 20% of the variation from ambient. By using a Cu tube (ordered) to replace the brass, the gradient should diminish on most of the sample. Also, 4 valves instead of one or two, should regulate the flow to the 4 segments of the sample for further gradient removal. See Figs. 2 and 14 through 19.

#### RESULTS

Figure 4 shows a change in sample length ( $\Delta L$ ) vs. change in temperature  $\Delta T$  for a 2.3" I.D. low CTE graphite/epoxy tube. Note the increasing hysteresis with increasing low temperature range. The slopes in runs #4 and #5 seem to double the previous slopes after venting to air to add a roller (Figure 1) under the tube. The small

slopes in heavy lines (P) are of other investigators after preparing similar samples with 50 cycles from  $-150^{\circ}\text{F}$  to  $+120^{\circ}\text{F}$ .

The runs immediately after opening the vacuum box to the air are indicated by the circle around the run number; see 1, 4, and 5 in Figure 4.

Although vertical hysteresis is up to  $10\mu\text{M}$ , the residual microcreep after overnight  $300^{\circ}\text{K}$  recovery is only  $1.6\mu$  (only  $\sim 2$  PPM). In other words, run 5B starts with the rod  $1.6\mu$  longer than it was in the start of run 5A. Certainly, the  $1.6\mu$  change did not result from ambient temperature being  $10^{\circ}\text{F}$  warmer the next day.

Such irregularities as the 2 x slope change indicate that all these TEC measurements seem only preliminary. Numerous data points were taken in the "turn around" parts of the curve to gauge the exact reversal shape.

Figure 5 shows preliminary sample length change ( $\Delta L$ ) as a function of temperature (T). Note the initial close agreement between runs 7, 8, and 9 during the first leg of the cycle. Hysteresis in varying amounts is seen in curves 9 and 11. The two C's indicate microcreep (up to  $10\mu$ ) of the sample measured on the next day on runs 9 and 11. Again the greater creep in run 11 (12 and 13 also) is attributed to the box having just been opened to air, as indicated by the circle about the number 11. The P's indicate the best slopes found by other investigators after 50 thermal cycles on similar G/E samples. The above slopes and  $\alpha$ 's are about 3 times larger than P except below  $0^{\circ}\text{F}$ , where our slopes are up to 30 x larger. The runs in Figure 5 prior to run 11 should be some of the best since the door had been closed for over a month and the sample had been thermally cycled about

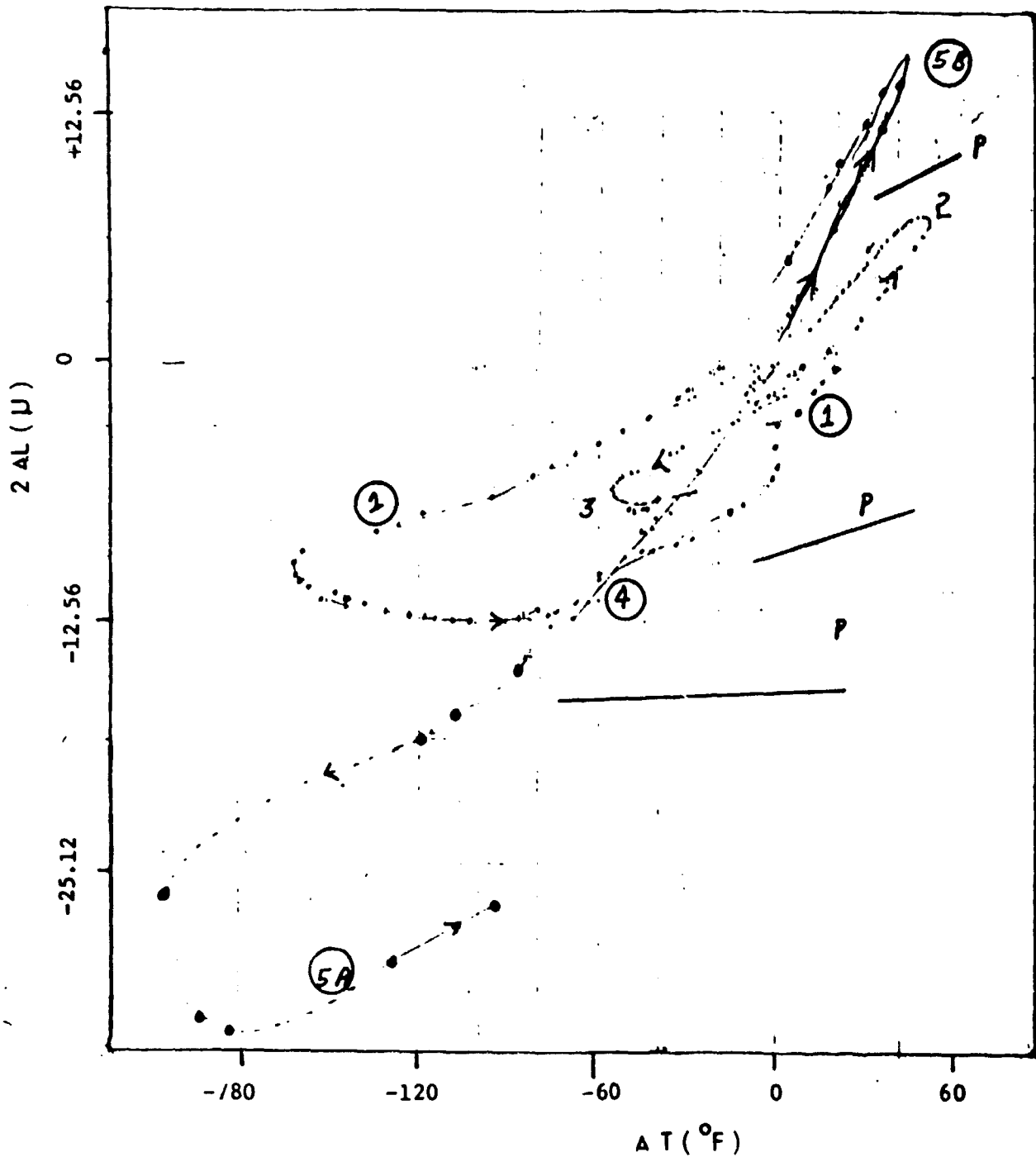


FIGURE 4

Figure 4.  $\Delta L$  vs.  $\Delta T$  for .9m Graphite Epoxy tube. Note the increasing hysteresis loops as the cycles push lower. The circles about the run numbers indicate the next run after the system was opened to air.



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Figure 5

Figure 5 is a continuation of Figure 4 showing  $\Delta L$  (in units of  $15.7 \text{ NM}$ ) . Run 9 should have been one of the better ones since it followed 25 cycles without exposure to air.

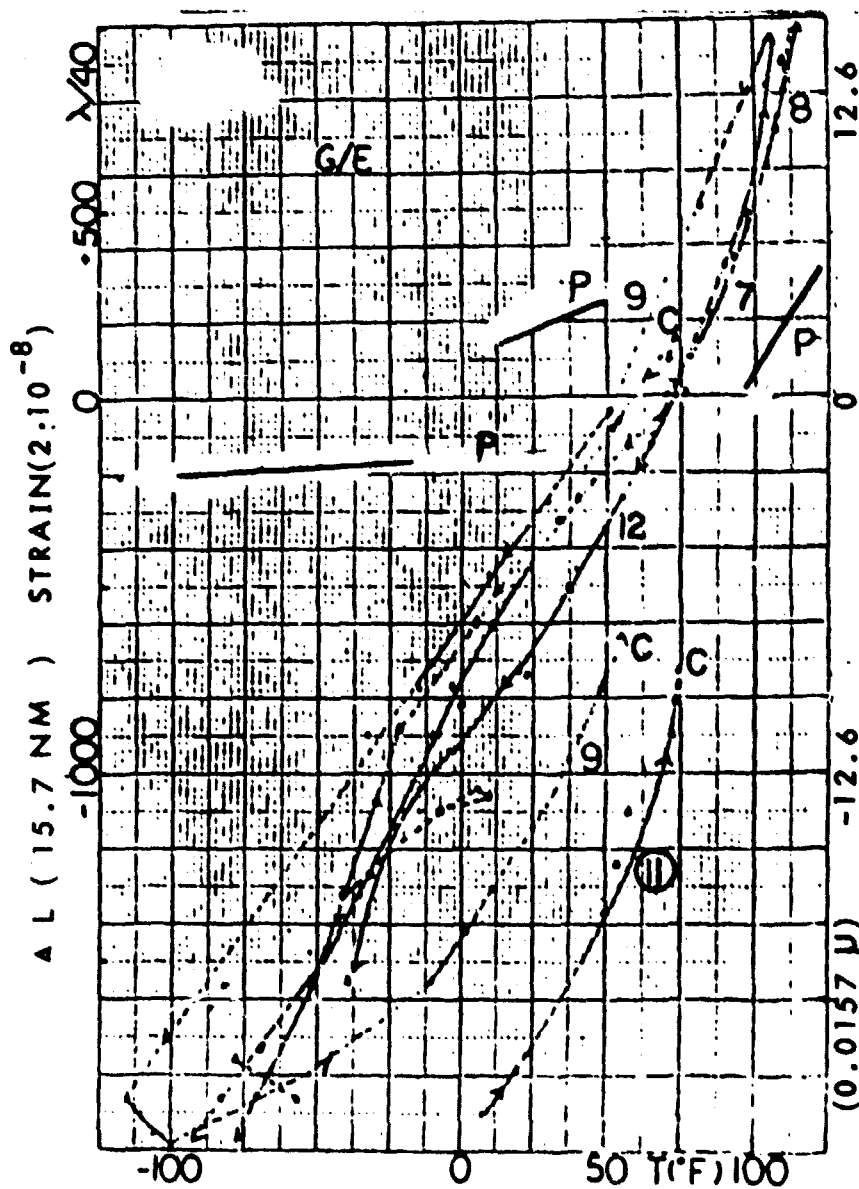
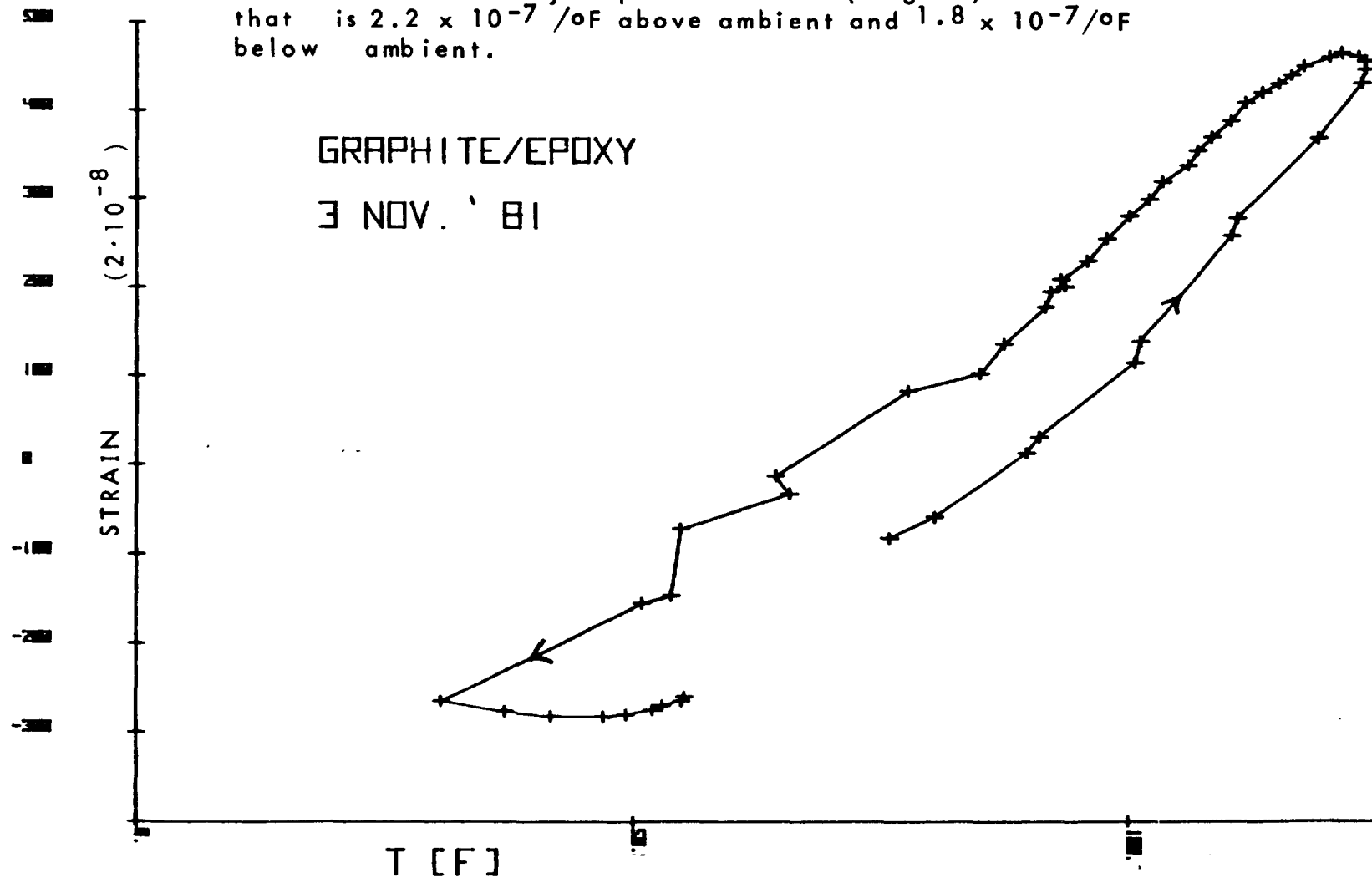


FIGURE 5

FIGURE 4A shows a single uncluttered Graphite Epoxy  
 $\Delta L$  vs.  $T$  run 2B just prior to run 3 (Fig. 4). Note  
 that is  $2.2 \times 10^{-7}$  / $^{\circ}\text{F}$  above ambient and  $1.8 \times 10^{-7}$  / $^{\circ}\text{F}$   
 below ambient.



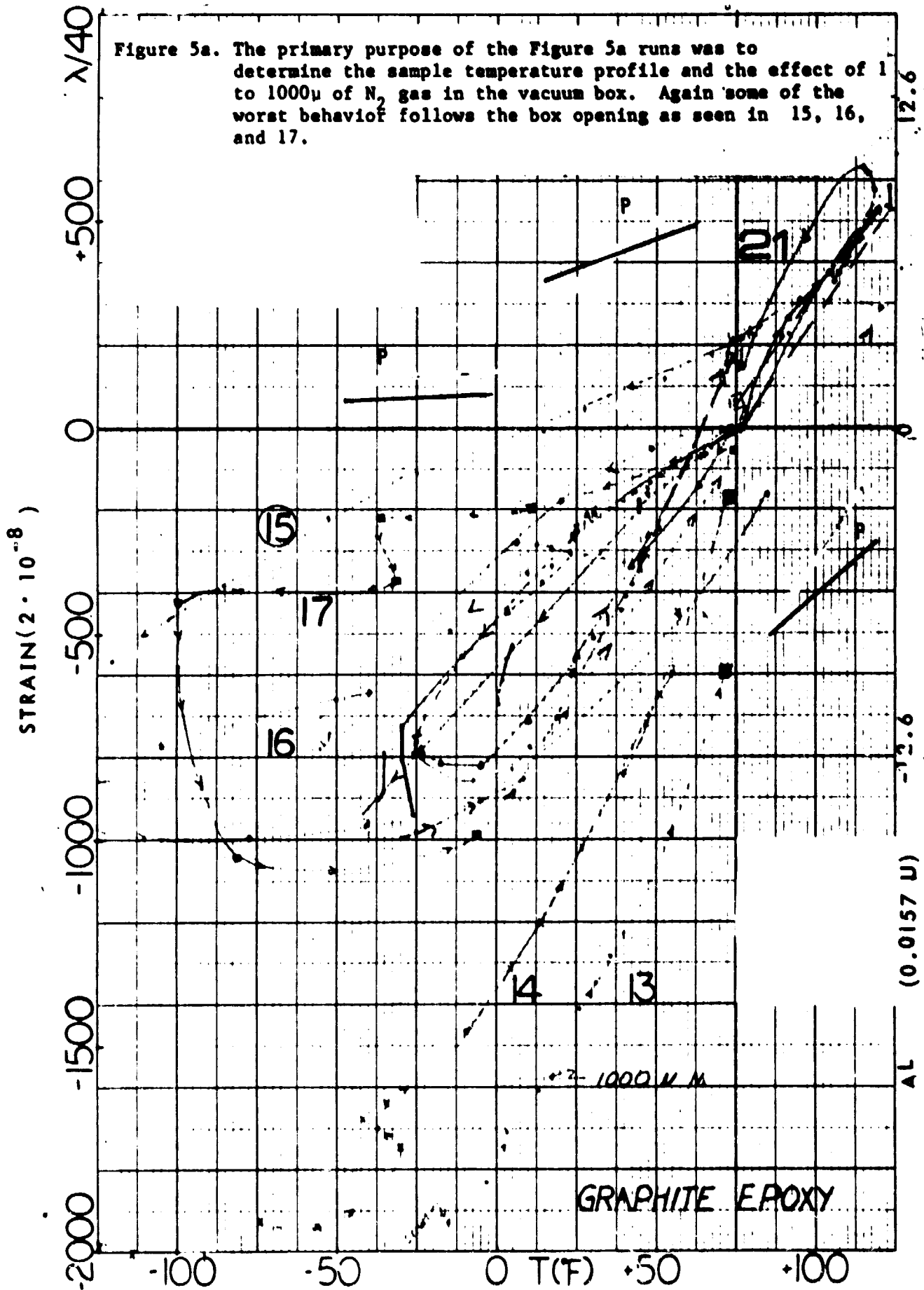


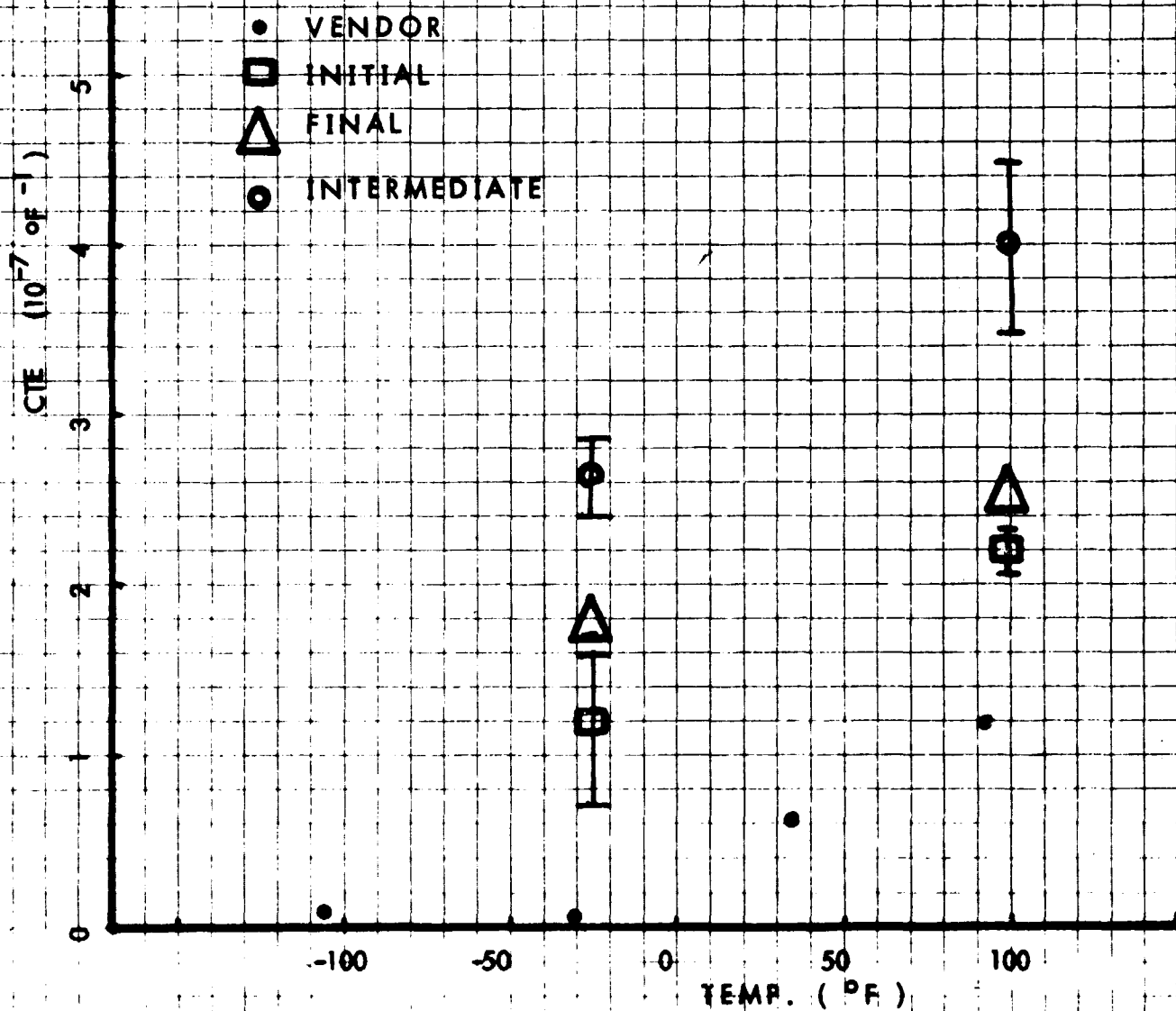
TABLE I  
Average Coefficient Thermal Expansion  
Graphite Epoxy Summary Table

<u><math>\bar{\alpha}(10^{-7}^{\circ}\text{F}^{-1})</math></u>	<u><math>\sigma</math></u>	<u>Figure</u>	<u>Runs</u>	<u>Time Period</u>
-100 $\rightarrow$ + 75°F	75 $\rightarrow$ 125°F			
$\alpha \pm \sigma$	$\alpha \pm \sigma$			
1.2 $\pm$ .5	2.2 $\pm$ .1	4	1,2B,3/2,2B,3	A( $\sim$ Nov.)
2.6 $\pm$ .2	4.0 $\pm$ .5	4 & 5	9,9B,11,12/5,7,8,9,9B	B( $\sim$ Dec.-Jan.)
1.8 $\pm$ .1	2.5	5A	18,19,14A/21	C( $\sim$ Feb.)
.09,.05,.63	1.2		Vendor	Aug.

Table I. Average values of coefficient of thermal expansion ( $\bar{\alpha}$ ) taken by averaging over several runs are shown. Note the surprisingly small average value of  $\sigma$  (15%) within each time period. The temperature dependence of  $\alpha$  is also less than expected. The smaller ideal vendor results are shown for comparison to ours.

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FIGURE 5 B

COEFFICIENT OF THERMAL EXPANSION  
OF GRAPHITE EPOXY

25 times. The 50 cycle goal without a door opening was not achieved. The sample strain may be obtained by multiplying the ordinate, expressed in integer (N) multiples of  $\lambda/40$ , by  $2 \times 10^{-8}$  in Figs. 5 through 7b. For example the 500 becomes 10 ppm.

The curves in Figure 5a have fewer points than those in Figure 4 since part of the Figure 5a objective is to generate the 50 thermal cycles before opening the box, to condition the G/E sample and thus reduce  $\alpha$ . Indeed in Figure 5a  $\alpha$  does drop by about 1.5 x in comparison to Figure 5.

Runs 12 and 13 are still bulging with hysteresis under the influence of the door opening (see Figure 7c also) prior to run 11. The 1000 $\mu$  of  $N_2$  gas pressure may have affected run 14.

The first leg of run 17 in Figure 5a has a very small (near ideal) slope (CTE) during the first part of the cycle, but as the whole cycle must be included in the average the CTE will be twice as large due to the hysteresis. The slightly smaller than Figure 5 slopes here may be due to the subsequent intervening thermal cycles. If the sample had the ideal CTE (see Table 1) with no hysteresis the curves would not drop below -200 ( $-3\mu$ ). Some of the runs are unnumbered for lack of space on the graph. Figure 4A shows curves plotted directly from magnetic data tapes through the HP 9815A desk top calculator. There is no smoothing as the line simply joins consecutive points. The arrows indicate the cycle direction.

### QUARTZ RESULTS

Figure 6 shows the expansion of .83M of Amersil quartz tube "A" as a function of temperature. Runs 1 and 2A give room temperature slopes, which are quite close to the accepted handbook average of  $.305 \times 10^{-6}/^{\circ}\text{F}$ . Run 2b and run 3 has about 1.4 x the expected slope for some reason. Thus  $\alpha$  for tube "A" is +20% above the handbook value. Notice the  $1.6\mu$  microcreep at the end of run 2. It is very doubtful that a  $7^{\circ}\text{F}$  error is causing the shift. The hysteresis ( $4\mu$  maximum measured vertically and  $20^{\circ}\text{F}$  measured horizontally) may be due to the outside mounted thermocouples leading (perhaps due to the proximity to the cooling and heating coils) the bulk sample volume temperature or due to frictional drag on the sample over the two knife edges. This drag, presumably, would have produced a horizontal flat clipping of the cycle extremes which was not observed.

Figures 6A and 6B were included to show details of a single run for quartz tube A. Note the clockwise hysteresis here in contrast to the G/E in Figure 4a, which looks like a  $20^{\circ}$  phase lag. The hysteresis is only below ambient temperature. In these early runs  $\dot{N}$  was neither recorded nor minimized before readings so the open loops could be due to the thermocouple lag. Figure 6a is just a replot from Figure 6 while Figure 6b is one of many unshown runs.

### RESULTS - QUARTZ

The second quartz [tube "B" Amersil Clear TO 8 Commercial 59 mm O.D.] system check results are shown in Figure 7 and 7b. The data look much better if one eliminates 13 March as being erratic as they eventually drifted 2 x off scale. The other points (included for diagnostic purposes) are the squares on the 75°F vertical which indicate the next morning readings after drifting to ambient (75°F) overnight. This overnight dotted line, connecting to the 75°F square, likewise should be removed as far as establishing a precision value of  $\alpha$  the CTE of quartz.

A straight line fit in the vicinity of room temperature gives a fairly precise estimate of  $\alpha$  of  $0.45 \times 10^{-6}/^{\circ}\text{F}$ . This is 50% larger than the handbook average value of  $\alpha$  for quartz. However, a value from the literature<sup>1</sup> (see Figure 8) on some of the best quartz available show typical variations of about 30%. Thus, one is unsure whether (a) the laser measuring system is reading 50% high or (b) this quartz has a 50% larger  $\alpha$ .

This overnight creep in Figure 7 was at first thought to be due to the glass like flow of quartz under slight pressure. Soft Cu shim rings were used between the invar rings' 3 screws and the quartz sample. Overnight quartz creep in Figure 7 ranges from  $6\mu$  to  $12\mu$ , a good bit higher, as expected, than the 3 to 4 hours creep in Figure 6. But since this quartz creep is essentially zero, (see Figure 7b), ( $<1\mu$ ) after a few cycles it is probably not a plastic flow.



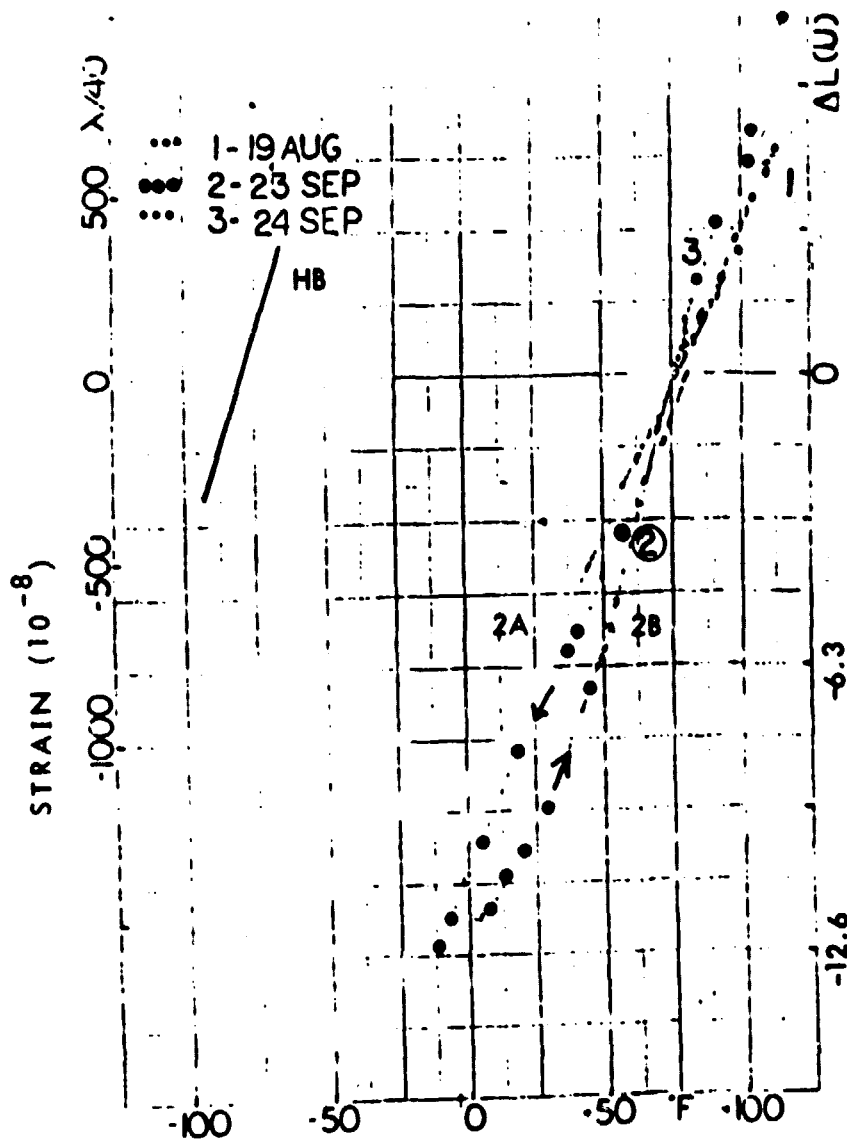


FIGURE 6 - QUARTZ TUBE EXPANSION VS.  
TEMPERATURE

Figure 6. First system check using quartz tube A. The ambient hand book slope is shown for comparison.

18 A

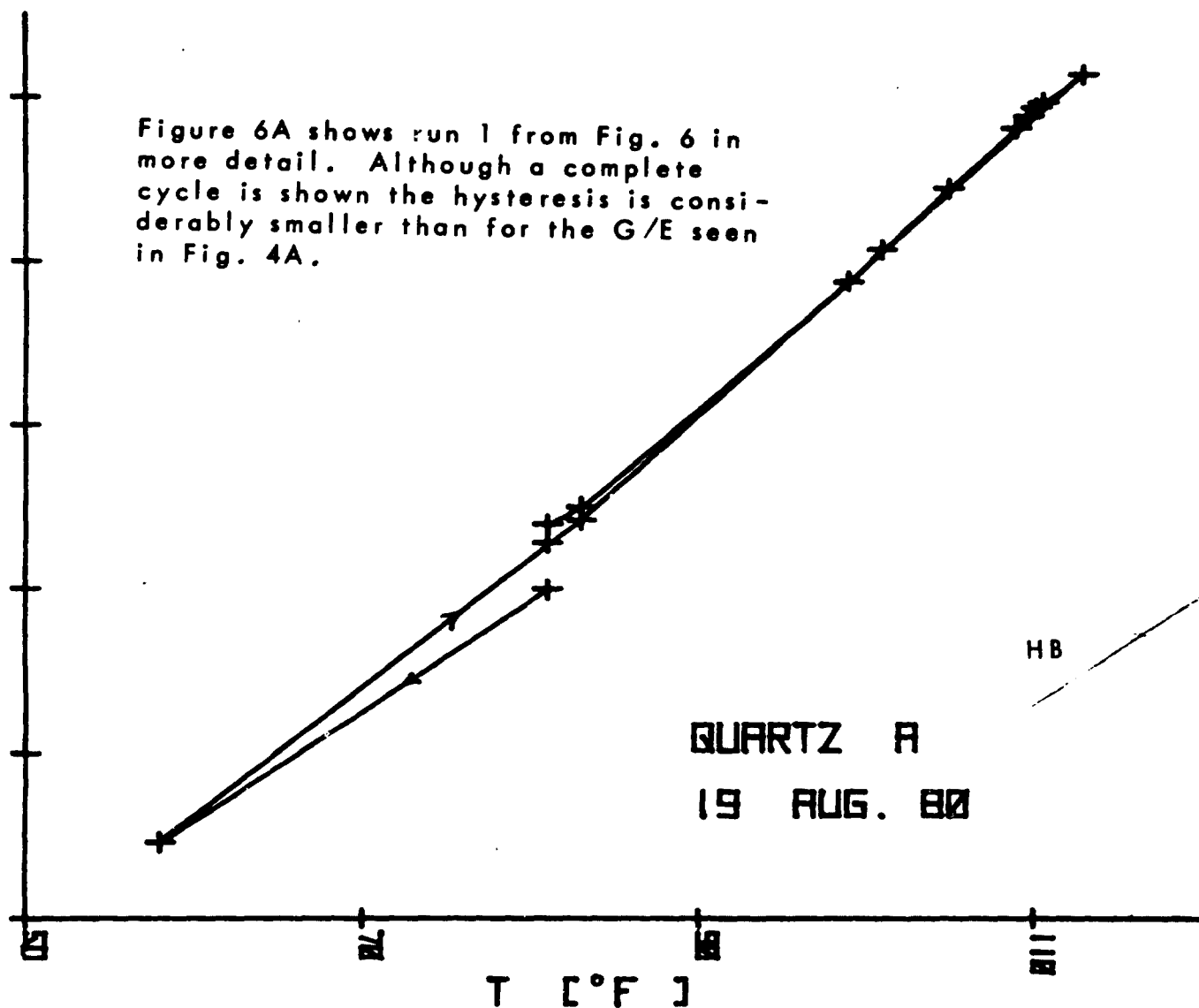
STRAIN

[  $10^{-8}$  . 2 . ]

$\Delta L$

[ 15.7 NM ]

Figure 6A shows run 1 from Fig. 6 in more detail. Although a complete cycle is shown the hysteresis is considerably smaller than for the G/E seen in Fig. 4A.



QUARTZ A  
19 AUG. 80

HB

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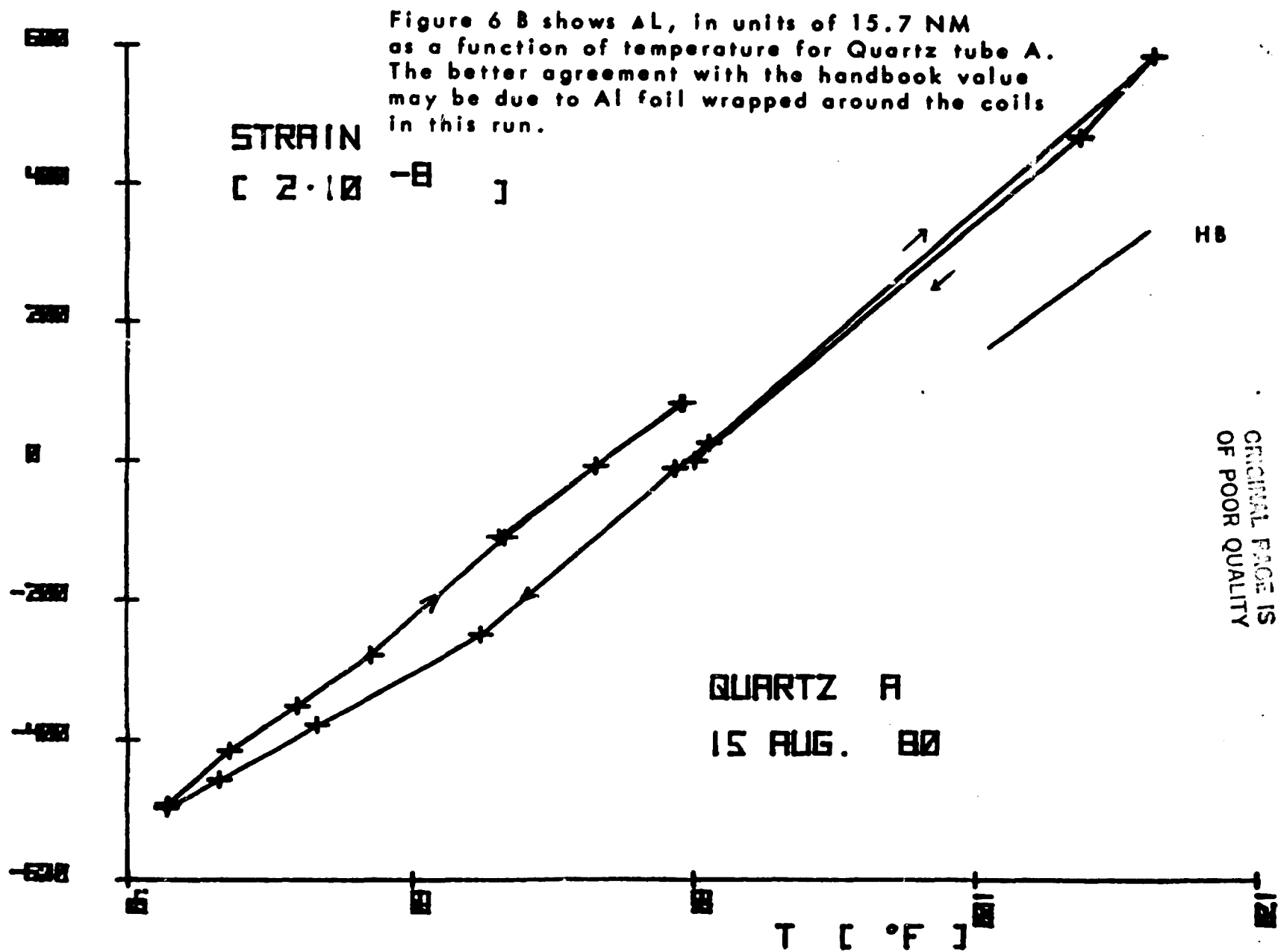


Figure 7 shows ten runs of thermal expansion vs. temperature for Quartz Tube B taken over a two week period. Runs 9, 3, and 1 are purposefully included to emphasize what can happen if the first runs after opening the box are not discarded. The squares at 75 °F indicate the next day's length after an overnight to ambient temperature.

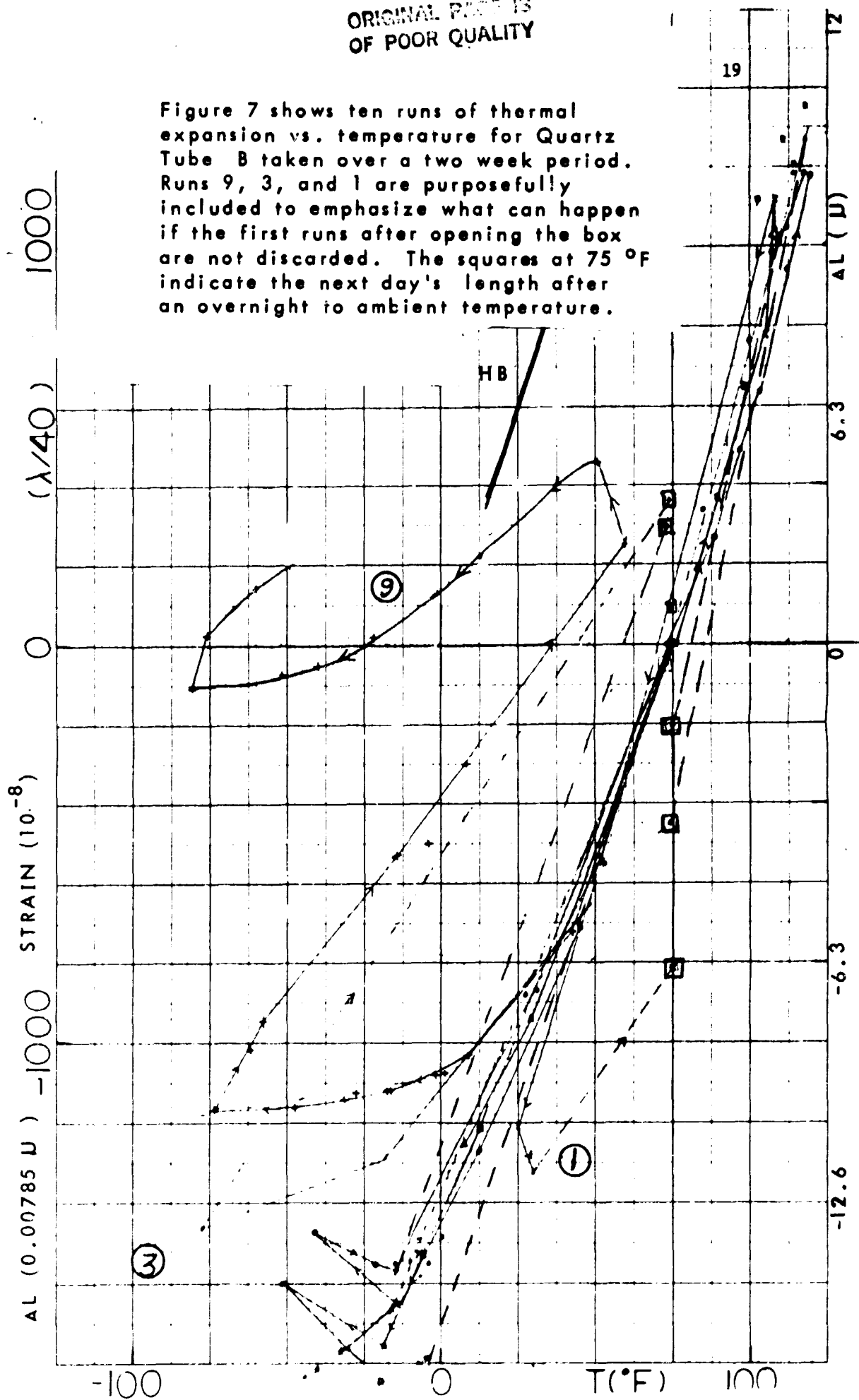


Figure 7b. Figure 7b is a clean-up of Figure 7; its clean, repeatable nature for 7 runs compared to the hysteresis in Figures 4 and 5 gives confidence in the CTE system when first runs after opening the box are deleted.

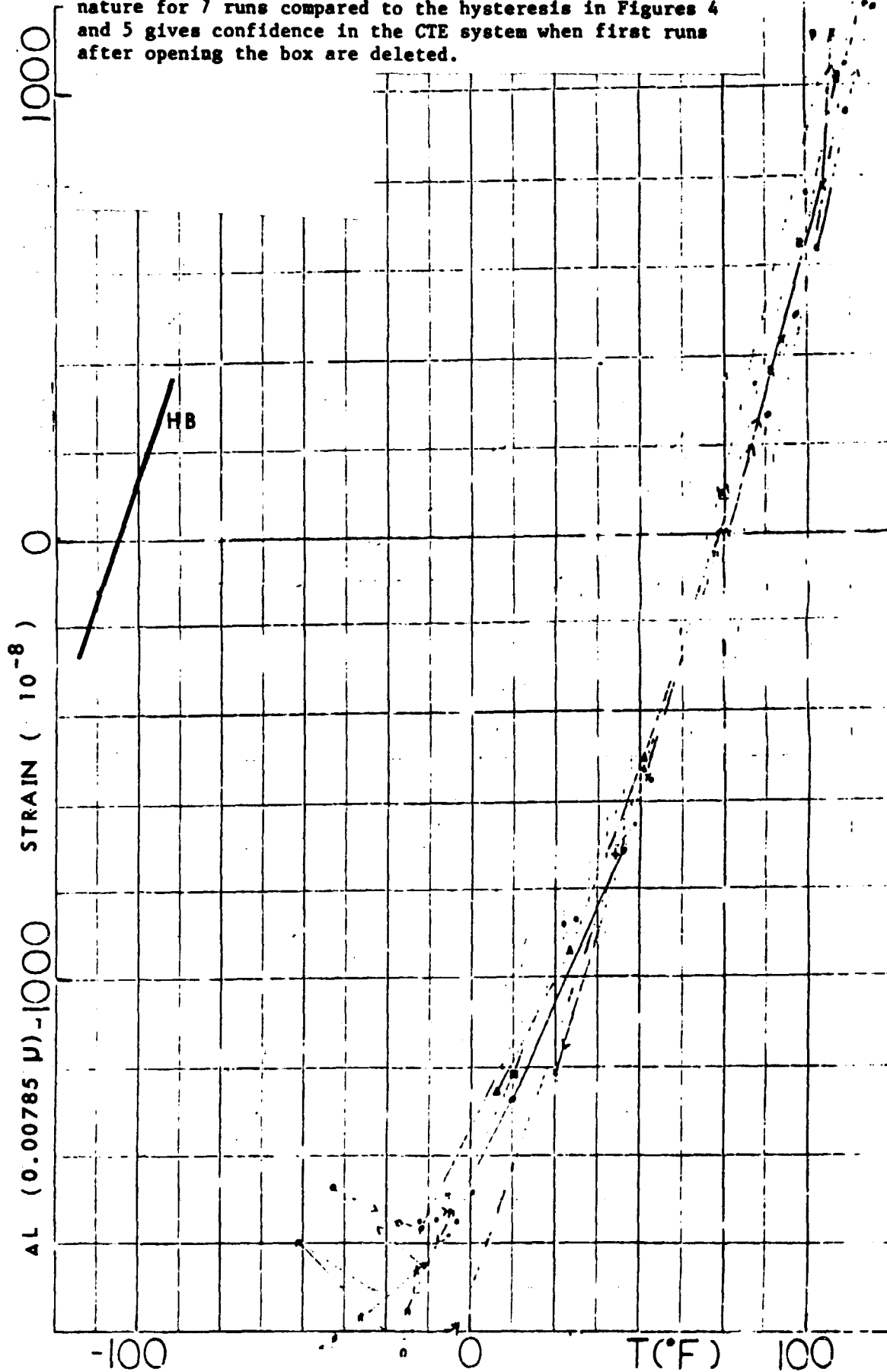


Figure 7 clearly shows again that the initial runs after opening the vacuum door, 1 , 3 , and 9 in this case, have the greatest divergence of all. They are dropped off in Figure 7b.

The quartz coefficient of thermal expansion ( $\alpha$ ) is clearly decreasing with temperature in Figure 7c; by taking the slope below room temperature by about 40°F, an  $\alpha$  close to the handbook average ( $3.05 \times 10^{-7}/^{\circ}\text{F}$ ) may be obtained.

If indeed the Laser Interferometer Measuring system is off +50% then the graphite epoxy expansion in Figure 5 would be only off by 1.5 x instead of the 3 x stated.

In Figure 6  $\alpha$  for a different quartz tube, "A", is averaging 30% higher than the handbook values. Accidental chipping and cracking of the quartz tube precluded the ideal continued use of the same quartz tube for the project duration. A second quartz diamond saw cut tube, B, was used later. It was fire polished on the cut to prevent the above cracking but was not furnace annealed.

Figure 7b shows also  $\Delta L$  (in units of  $\lambda/40 = .0157\mu$ ) vs. T for Quartz Tube "B". Figure 7b is a cleanup of Figure 7 in which the three first cycle curves, 9 , 3 , and 1 were removed along with some overnight creep points. Seven runs for a 2-week period are seen. The reproducibility and very small ( $<1\mu$ ) hysteresis of these quartz curves provide an "acid" test to the precision and stability of the CTE system. However, since this slope is about 50% larger than the average ambient H.B. slope for quartz on the left, no claim about absolute accuracy is made from this figure. The term "acid test" is used since quartz has a very low thermal conductivity (exaggerating thermal gradients and relaxation times) and a CTE which approaches zero.

The following consideration could explain the 50% high CTE of our quartz (fused silica) tube "B". The Amersil commercial quartz spec. T08 quartz tube (because of its .018% O-H spec. sheet O-H content) probably falls into the type II category<sup>5</sup> ( $\sim .04\%$  O-H content). Bruckner reports<sup>5</sup> (see Figure 9)  $\alpha$  rising from about 60 to  $\sim 80 \times 10^{-8}/^{\circ}\text{C}$  for both types I and II quartz as the fictive temperature rises from about 1000°C to about 1500°C. Our one end flame polishing of B (after diamond saw cutting) without subsequent annealing could have given a higher fictive temperature to that end. A 1145°C anneal schedule may be desirable next time. The larger  $\alpha$  may be more like that expected of type III quartz ( $\sim 1000$  PPM O-H content). See Figure 9.

A second indication of higher fictive temperature is noted in quartz tube B in Figure 7. The temperature of zero  $\alpha$  appears to have shifted  $\sim 90^{\circ}\text{F}$  higher (to about  $-15^{\circ}\text{F}$ ) than the zero  $\alpha$  value in Figure 8 (200K or  $-1.07^{\circ}\text{F}$ ). Buckner<sup>5</sup> (his Figure 11) also finds a  $+50^{\circ}\text{K}$  ( $+90^{\circ}\text{F}$ ) shift in the zero  $\alpha$  temperature as the fictive temperature is increased from about 1000°C to 1500°C. Both Figure 7 and Figure 11 from Wolff show precisely  $33 \pm 1$  and 32 PPM quartz contraction respectively from ambient to the zero  $\alpha$  point.

Least-mean-square fits to a polynomial of the  $\Delta L/L$  vs.  $T$  data provided the temperature dependence of  $\alpha$  for quartz tube B. The Texas Instruments T.I. 59 calculator with Mathematics Specialty Packet program # 398010A was used.

With considerable hysteresis a tear-drop shaped curve may have been a better choice, e.g., one leaf of an  $n$  leaf rose whose equation is given:  $r = a \sin(n\theta)$  where  $n$  is an odd integer decreasing with the fullness of the tear drop.

### MICROCREEP VS. CYCLES

As seen in Figure 7C the overnight microcreep in both the G/E and in the quartz "B" was about  $12\mu$  in the run after opening the box door but decreased gradually (roughly exponentially) for several days leveling out in about one week to  $1\mu$  creep per cycle.

The most persistent microcreep ( $\sim 2\mu$  after a week) was after the box was left open for a (14-15 Feb) 24-hour period as seen in Figure 7c.

The fact that the microcreep occurs almost identically in the quartz "B" and G/E sample implies that it may be apparatus-related. But others also find considerable (Figure 11) creep which decreases after a few cycles for G/E<sup>3,6</sup> and quartz.

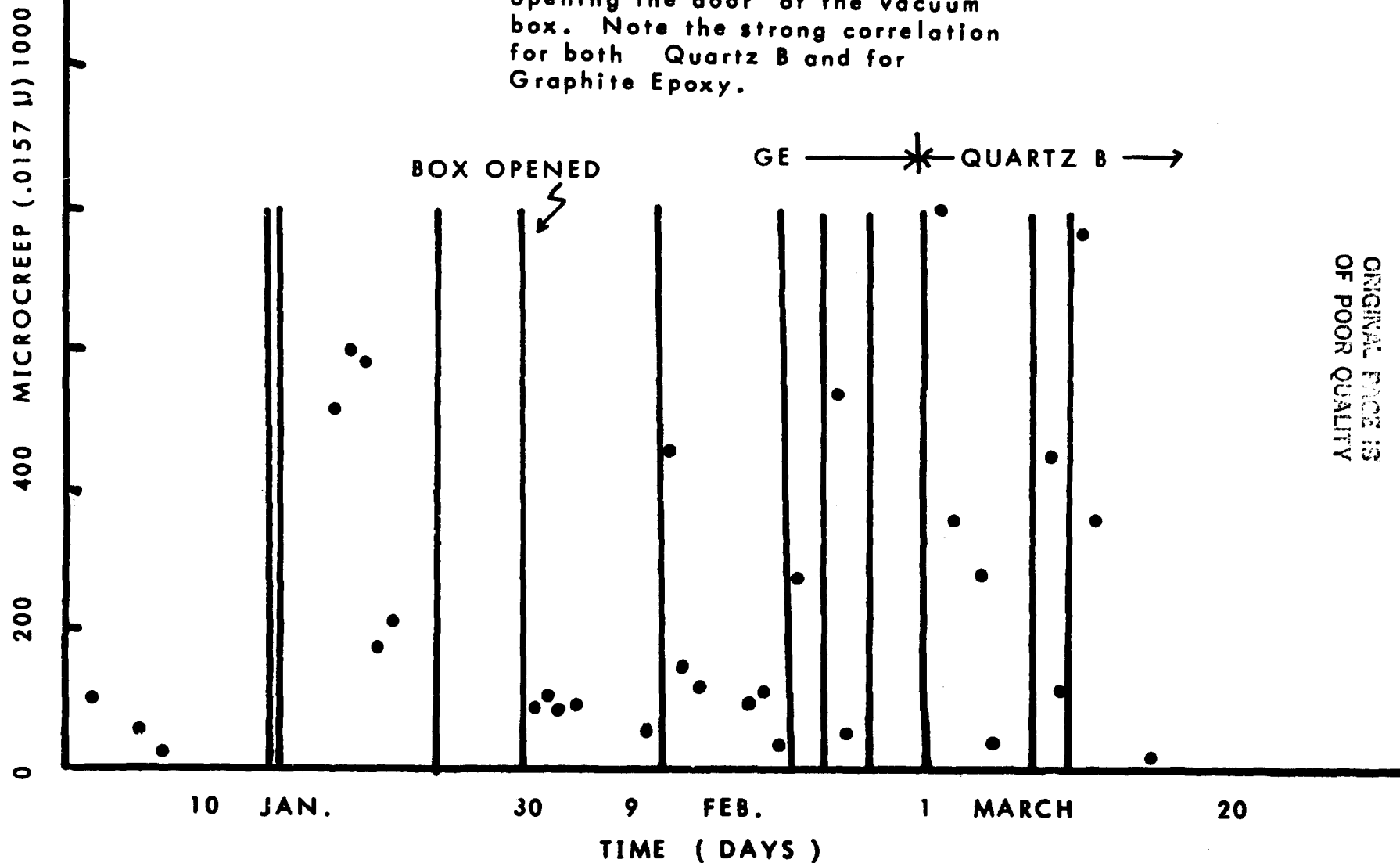
Most other investigators do not use the vacuum thus a direct comparison is not possible. Is it the venting to air or the accompanying sample support adjustment which reintroduces creep? Figure 7c does not indicate whether the vacuum, pure time, or thermal cycle cured the creep.

Figures 12 through 19 show the temperature profile of the G/E sample at various temperatures during both heating and cooling. Figure 12 shows that the sample temperature is much more uniform in the active region between the mirrors upon cooling than upon subsequent rewarming. Thus a greater confidence is suggested on all cooling curves in Figures 4, 5, 6, and 7.

The greater  $N_2$  gas pressures in Figures 13, 14, and 16 increased thermal conductivity but did not seem to help the temperature profile (except perhaps in Figure 16) when compared to the profiles under vacuum ( $< 1\mu$  Hg.) in Figures 12, 15, 17, 18, and 19. The six thermocouples were mounted on the G/E tube in Figures 12-17; 3, 6, 7, 1, 4, and 2.



Figure 7 C shows a plot of overnight microcreep as a function of time in days. The eleven vertical lines indicate the day of opening the door of the vacuum box. Note the strong correlation for both Quartz B and for Graphite Epoxy.



respectively were not equispaced but placed at 18 5/8, 15 3/4, 12 5/8, 8 1/2, 5 1/4, and 2 5/8 inches respectively from the back end of the sample. T.C. #3 was at the center and T.C. #2 was at the mirror mount.

#### PRESSURE DEPENDENCE OF LASER DISPLAY

Since a few microns of gas pressure could conceivably be used to smooth the temperature profile, a check on the effect of pressure on the laser display was made for comparison to the theoretical value

$$\frac{(n-1)2L}{760 \text{ mm}} = \frac{0.66\mu}{1 \text{ mm Hg.}}$$

The box N<sub>2</sub> pressure was cycled ten times, from less than 1 μ to various values less than 1000μ; the resulting pressure derivative of the (182 cm) optical path was found to be 0.66 ± .02μ/mm Hg. of N<sub>2</sub>.

The absolute error in the above quantity is only +4% while the standard deviation is only 3%. These results provide a welcome test to most of the overall system (including the T. C. gauge) except for the thermocouples.

#### ERROR

In measuring an ultra small quantity, one must be very careful to identify and compensate for all sources of error. Confidence is usually generated by the reproducibility of the experimental measurement. Here, with considerable initial G/E sample hysteresis expected in ΔL vs. T, one was initially concerned that some of the nonreproducibility may be apparatus-caused. The last system check using quartz had been about four months earlier. This laser CTE system had been modified and continually upgraded since calibration; thus, all ΔL vs. T results were taken as tentative until the second quartz check. (Seen in Figure 7b.)

The temperature profile on the last quarter of the sample has now been measured at ten different center temperatures. The effective

ORIGINAL PAGE IS  
OF POOR QUALITY

T. A. HAHN and R. K. KIRBY

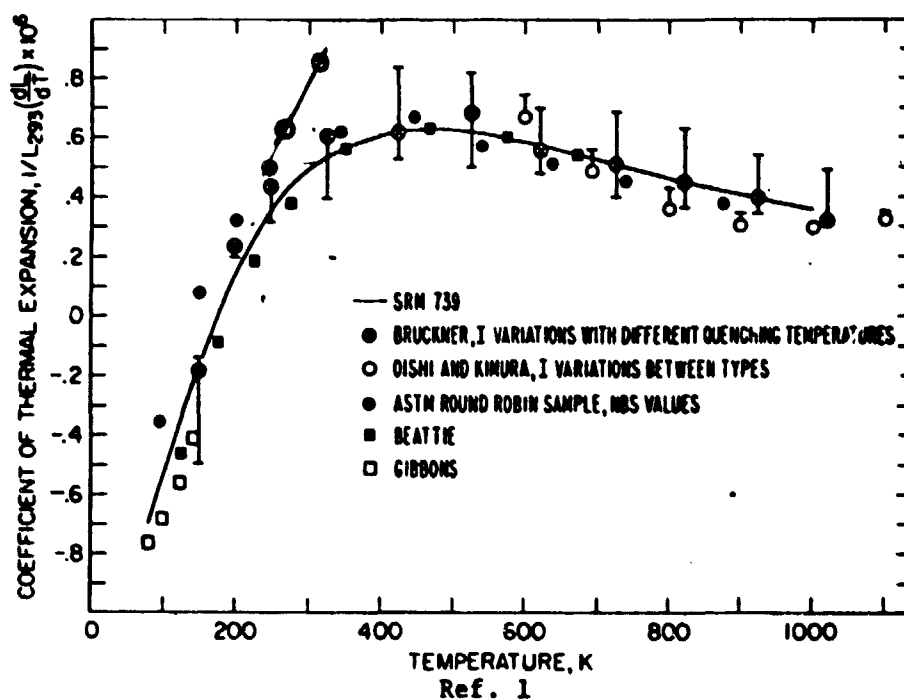


Fig. 8 Comparison of the coefficients of expansion of fused silica from different investigators.

—○—○—

**PRESENT**

# CHARACTERISTICS OF POOR QUALITY

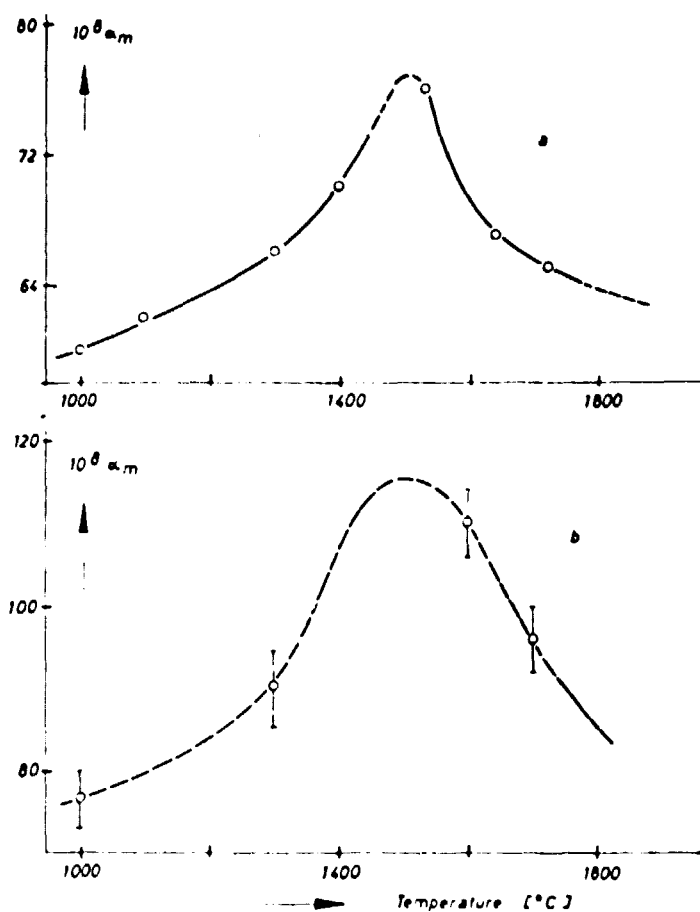


Fig. 9 Thermal expansion coefficient of type III (a), and III (b), silica glasses as a function of fictive temperature.

Bruckner (Ref. 5)

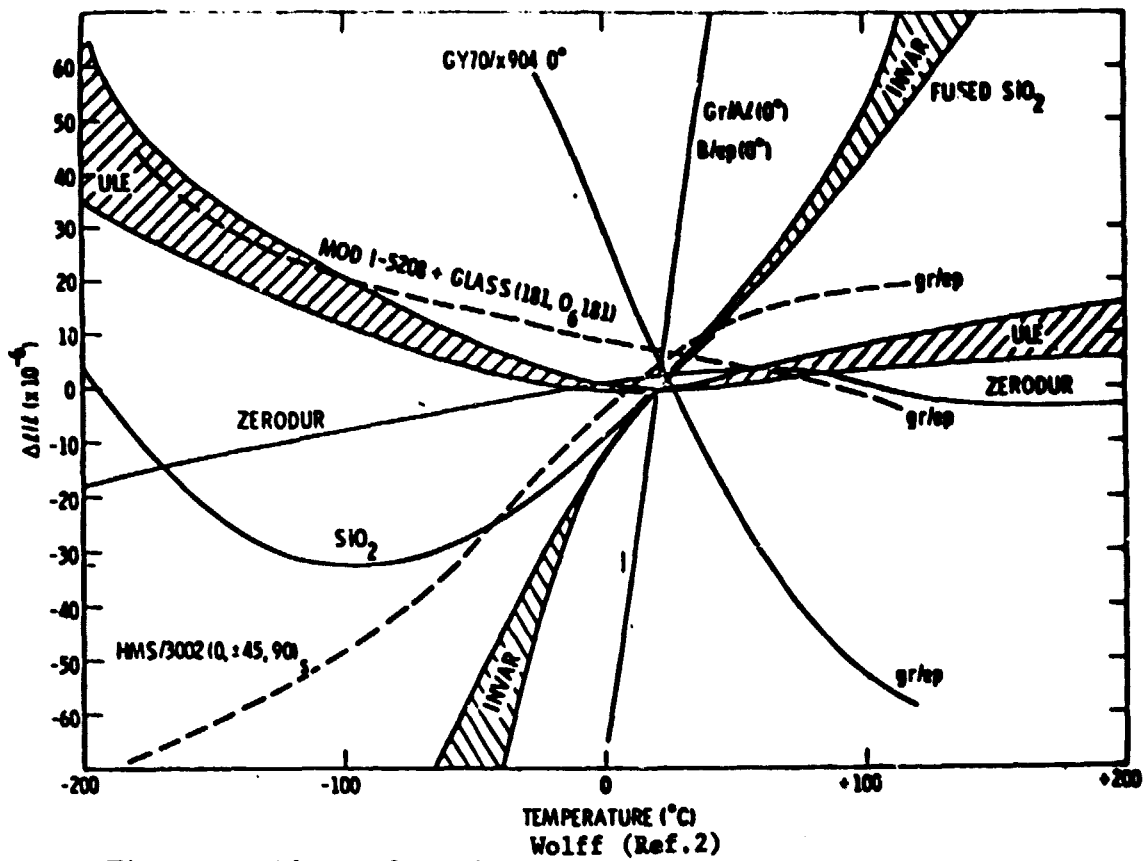


Figure 10 Thermal strain for various low expansion materials

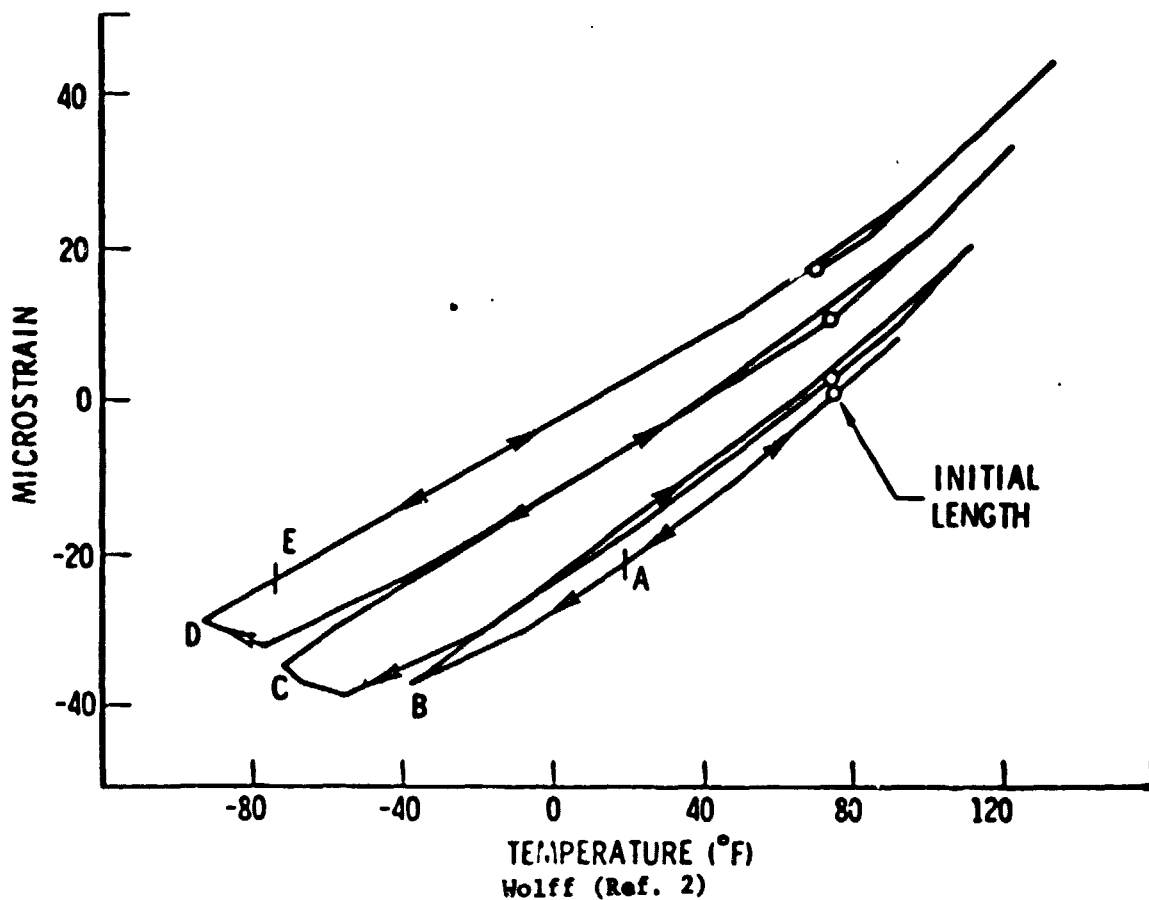


Figure 11 Length changes of a Gr/ep Tube due to thermal cycling

heated or cooled length may be slightly less than the .83 M used. At  $-70^{\circ}\text{F}$  the outside 1/4 of the G/E sample was found to average  $30^{\circ}\text{F}$  warmer than the center giving a  $\sim -5\%$  error in  $\Delta L$ , which results in  $\sim -5\%$  error in  $\bar{\alpha} \equiv [L(75^{\circ}\text{F}) - L(-70^{\circ}\text{F})]/145^{\circ}\text{F}$ . A  $+ 5\%$  or  $10\%$  correction may be easily made if  $\alpha = \text{const.}$ , but since  $\alpha$  is not constant over the whole temperature range, the temperature profile should be further equalized.

One may hypothesize that the G/E rod actually has near zero  $\alpha$  below a certain temperature but that this near zero  $\alpha$  was missed due to contractions near the two warmer ends. If in fact the sample of G/E has the ideal  $\Delta L$  vs.  $\bar{T}$  slopes indicated by the P's in Figure 5, then the rod's total contraction from  $120^{\circ}\text{F}$  to  $-100^{\circ}\text{F}$  would have been only  $\sim 1/4$  of the actual  $\Delta L$  found in Figure 5. Thus the above hypothesis is ruled out.

Cosmetically our  $\Delta L$  vs.  $\bar{T}$  curves and points look rather rough in comparison to some of the smoothed literature where the data points have been either erased or omitted. Our  $\bar{T}$  is not a time average or an average over several runs but is only a spatial average over several sample thermocouples since the sample is rather long.

Earlier the possibility was raised that some of the drift and microcreep was in the electronics. Since the  $\mu$  creep vanished in a few cycles (see Figure 7c) (even with the over the weekend cases) the electronics were cleared of suspicion.

## DISCUSSION

### Samples

An internal report indicates that even after 50 cycles CTE measurements could not be made on some of these Graphite Epoxy samples

due to gross hysteresis of  $\Delta L$  vs.  $T$ . Perhaps one of the above rejects was delivered by mistake. In fact, one person indicated that the GE strut should have mirror mount holes from previous CTE measurements.

Perhaps the hysteresis returned with age of the strut. Perhaps the  $1\mu$  vacuum enhanced the hysteresis. Feund<sup>3</sup> found sample length changes with a vacuum and thus allowed the sample to stabilize 16 hours before testing. Perhaps the cryogenic  $LN_2$  line entering the box provided some cyclic water absorption and later emission which influenced the sample length.

Eselun Neubert and Wolff<sup>6</sup> performed recent  $\Delta L$  vs.  $T$  opto-acoustic measurements on two 3' Fiberite Graphite Epoxy tubes GY 70/934. They found the sample length change  $\Delta L$  in terms of

$$\Delta L = \alpha \Delta T + \beta \Delta M (^\circ/\omega) + \gamma \Delta T;$$

where  $\alpha \Delta T$  is the conventional displacement due to thermal expansion,  $\beta \Delta M (^\circ/\omega)$  is the displacement due to moisture absorption and  $\gamma \Delta T$  is an irreversible displacement caused by microcracking below the onset temperature.

Their finding for the  $\pm \bar{+} 30$  tube was:

$$\text{axial } B_c (\% M^{-1}) = 337 \times 10^{-6}$$

$$\alpha (10^{-6} \text{ } ^\circ F^{-1}) = + 2.4.$$

For the  $0/\pm 60/6$  tube they found:

$$\text{axial } B_c \% M^{-1} = 98 \times 10^{-6}$$

$$\alpha (10^{-6} \text{ } ^\circ F^{-1}) = -.4$$

$$\gamma (10^{-6} \text{ } ^\circ F^{-1}) = .123$$

$$\text{Twist (arc sec/} ^\circ F) = 3.6$$

The fractional length change was 84 ppm due to absorption of 0.86% (wt.) water, 20 ppm due to matrix microcracking down to  $-184^{\circ}\text{F}$ , 80 ppm due to a temperature change of  $200^{\circ}\text{F}$ .

So none of the  $\alpha$ ,  $\beta$ , or  $\gamma$  contributions are negligible.

Their drying procedure after exposure to 68% humidity was to place the sample in a room temperature vacuum for one week while monitoring the weight, and to raise the sample temperature to  $150^{\circ}\text{F}$  to finish drying out and then thermally cycle from  $75^{\circ}\text{F}$  to  $-200^{\circ}\text{F}$  at  $6.4^{\circ}\text{F}/\text{min}$  while monitoring the strain.

We, like Wolff<sup>2</sup>, find large initial hysteresis below  $-60^{\circ}\text{F}$  in Graphite Epoxy. Wolff's initial (45° wrap) G/E curves (see Figure 11) open horizontally to a width of  $\sim 40^{\circ}\text{F}$ . In fact both G/E hysteresis curves cycle counterclockwise and ours (including most quartz) cycle counterclockwise. Why? See Figures 4, 5, and 11. Most others<sup>3,6</sup> also find similar initial hysteresis in G/E; however, ours is more persistent.

An open C.C.W. hysteresis loop is expected if a frictional drag is restricting an elastic samples expansion. Although such extensive drag was eliminated by the roller, an intrinsic component may be present in the plastic epoxy.

The counterclockwise cycles could be due to the thermocouple leading the true mean sample temperature (and vice versa for the C. W. cycle).

The 3x to 30x factor by which the G/E CTE is larger than that found by previous experimenters (see Figure 4) is not surprising since our G/E sample has not been stabilized by 50 thermal cycles. Figures 5 and 5a should have a reduced hysteresis and a reduced  $\alpha$  because runs 9 and 10



have undergone  $\sim 25$  thermal cycles since opening the door to the atmosphere. In fact, the 2x increase in CTE, upon addition of the roller, may have been due to the simultaneous venting of the system to room air.  $\alpha$  seems to be rising each month in spite of the fact that each run constitutes a cycle which should tend to lower  $\alpha$ . The system is usually vented to room air for various reasons about once per week, possibly wiping out any reduction in  $\alpha$  due to cycling. Perhaps the 50 cycles should be done within one or two days.

An uncycled 1 ft. section cut off the end of the G/E tube has been stored on the lab shelf. Its  $\alpha$  should be measured with cycles only above 32°F to avoid the possibility of any water freezing complications which may have caused the gradually increasing  $\alpha$ .

There may be some signs of cycle induced CTE ( $\alpha$ ) reductions in the large flat-bottom cycles such as run 1 of Figure 4 where  $\alpha \sim 5 \times 10^{-8}/^{\circ}\text{F}$ .

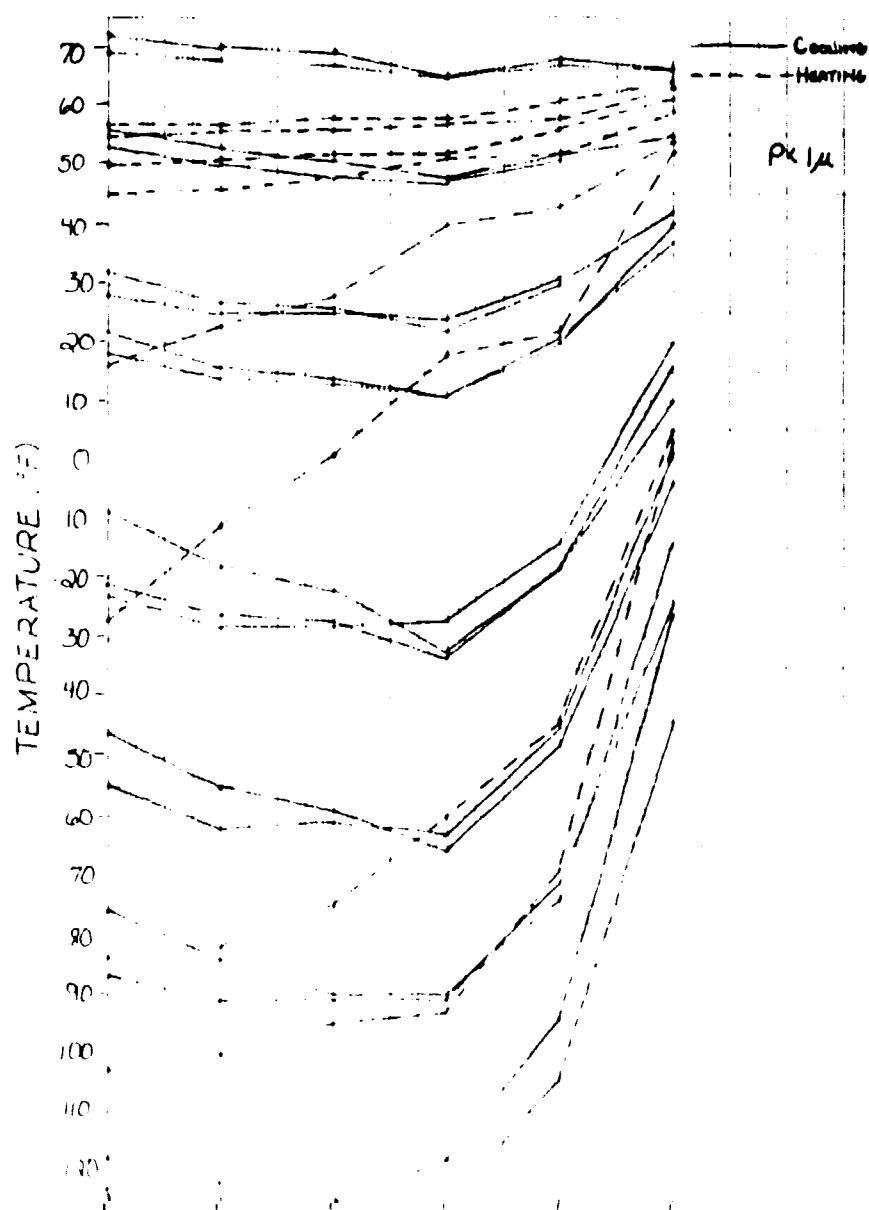
Our data points do seem to have some scatter in comparison to the smoothed and averaged lines in the literature.<sup>1</sup> However with novel systems and one of a kind materials it is best not to lose any information in the averaging and smoothing process.

Wolff<sup>4</sup> also is seeking a 1M long calibration standard for his laser interferometer and finding that even the best grade of Type I quartz has sample to sample variations below 300°K. His type I quartz is a good full cut above ours in quality. Brucher<sup>5</sup> finds early types I and II quartz to have the same behavior in  $\Delta L$  vs.  $T$ .

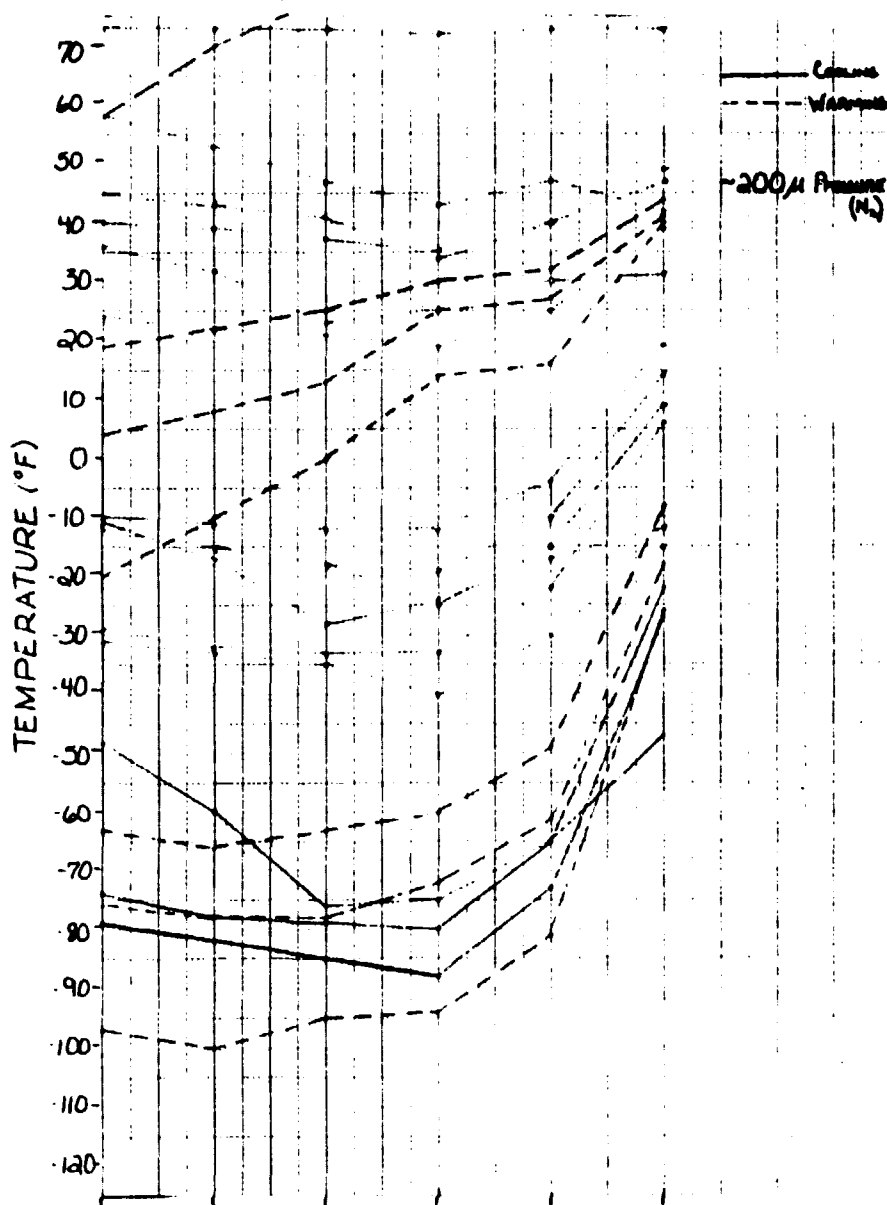
# TEMPERATURE GRADIENT OF POOL QUARTZ

Figure 12 - Thermal profiles of back half of G/E  
sample at  $P < 1 \mu H_g$

TEMPERATURE GRADIENT ON BACK HALF G/E



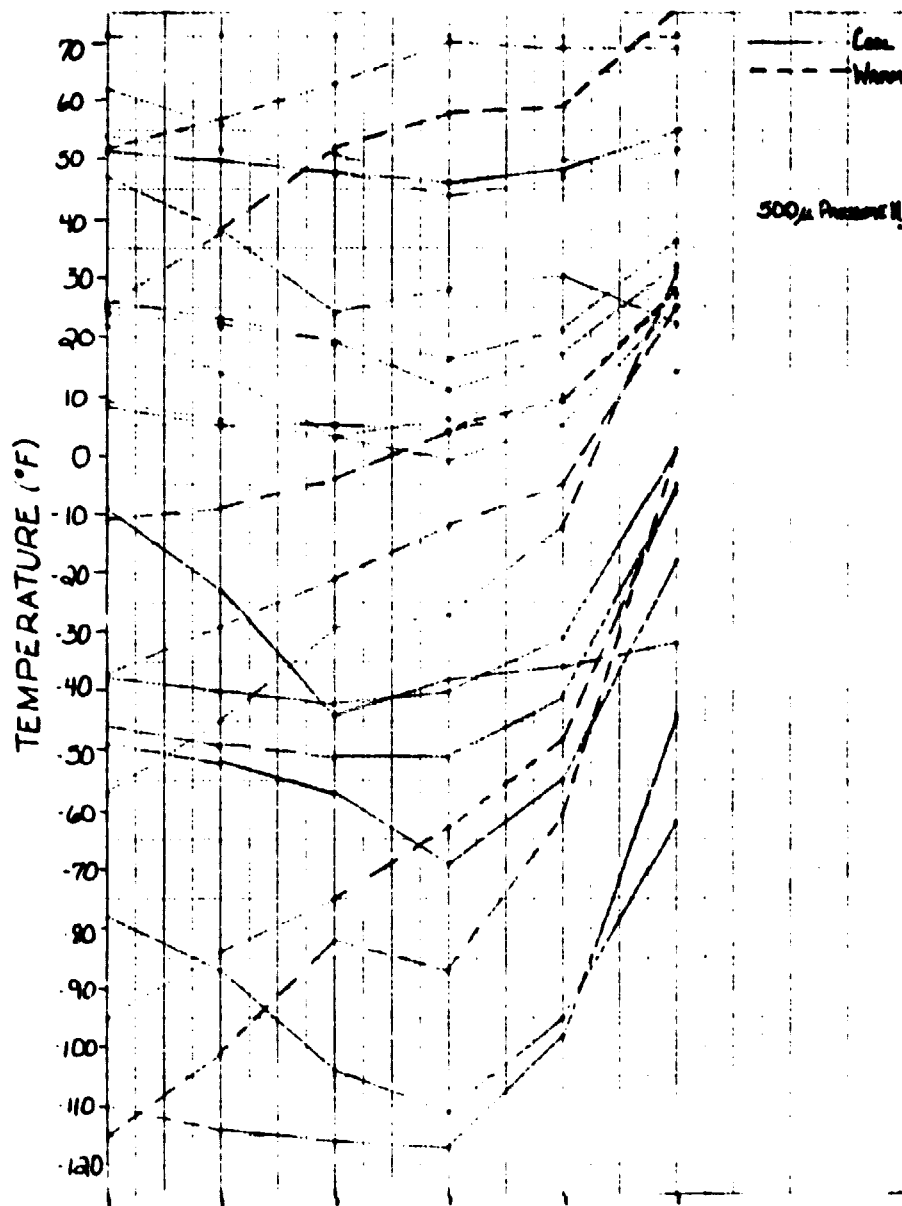
APPROXIMATE DISTANCE ALONG SAMPLE



APPROXIMATE DISTANCE ALONG SAMPLE

Figure 13 - Thermal profile along the back (i.e. away from the laser) half of the Graphite Epoxy sample with a trial atmosphere of  $200 \mu (H_2)$ .

## TEMPERATURE GRADIENT ON BACK HALF G/E



APPROXIMATE DISTANCE ALONG SAMPLE

Figure 14 - Thermal profile along the back (i.e. away from the laser) half of the Graphite Epoxy sample with a trial atmosphere of  $500 \mu (\text{H}_2)$ .

GRAPHITE  
OF EPOXY

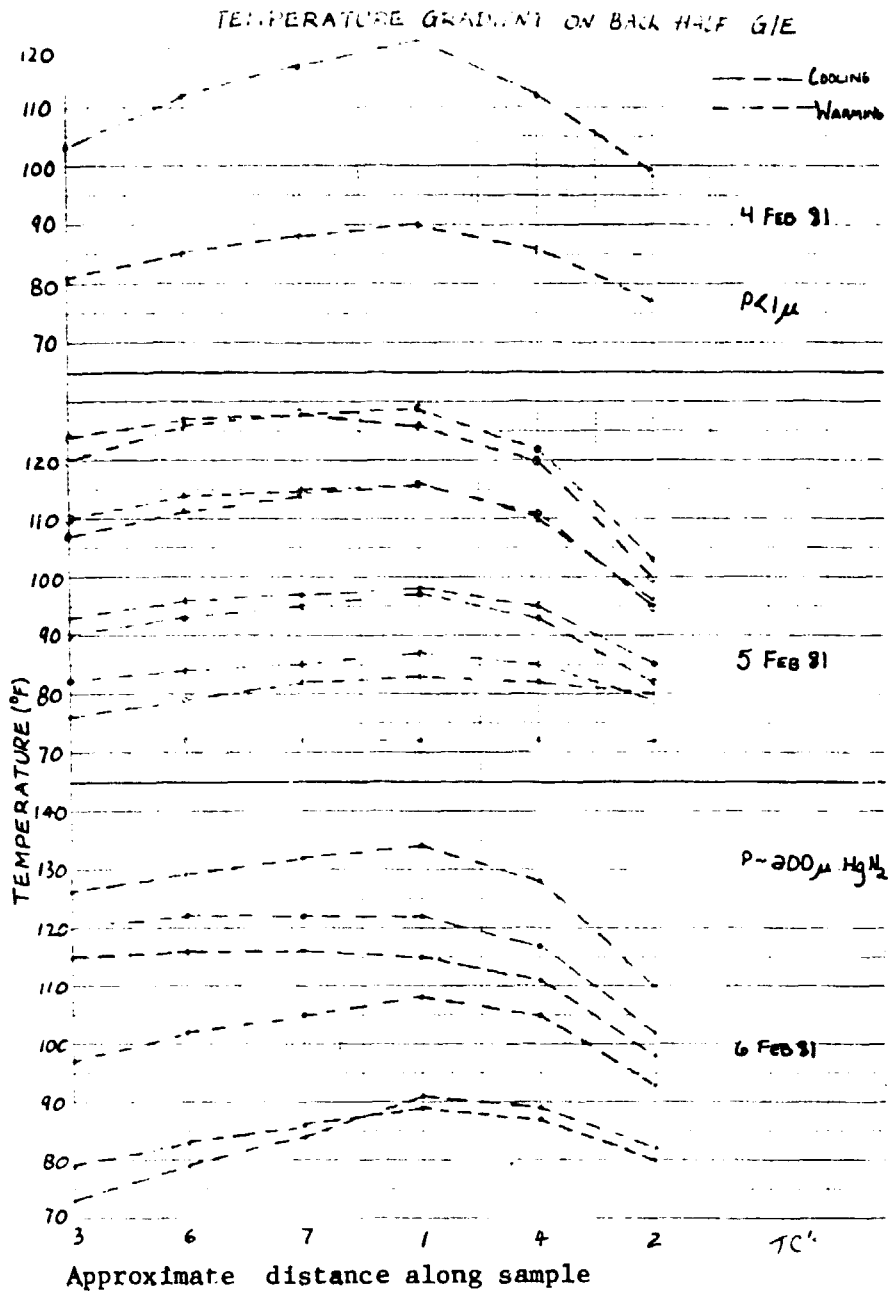
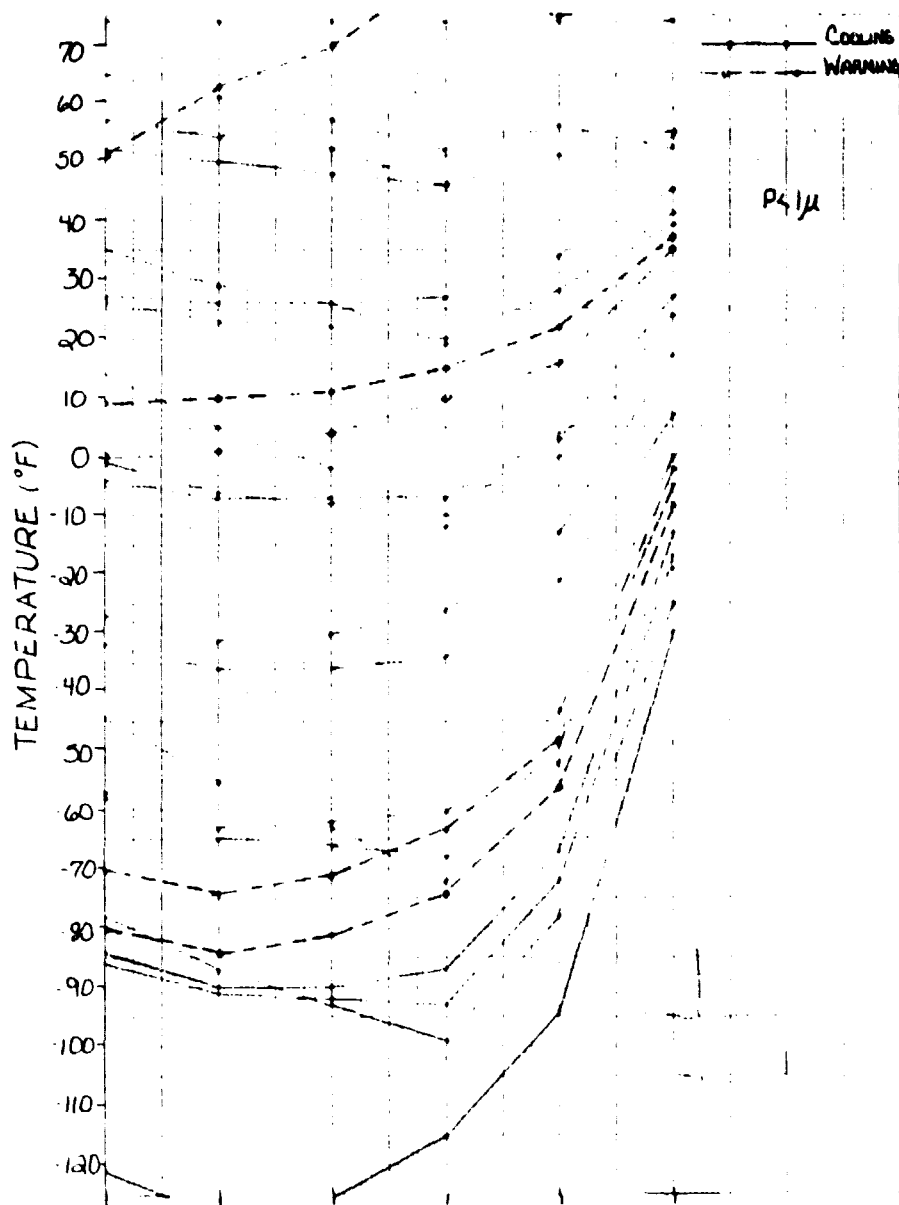
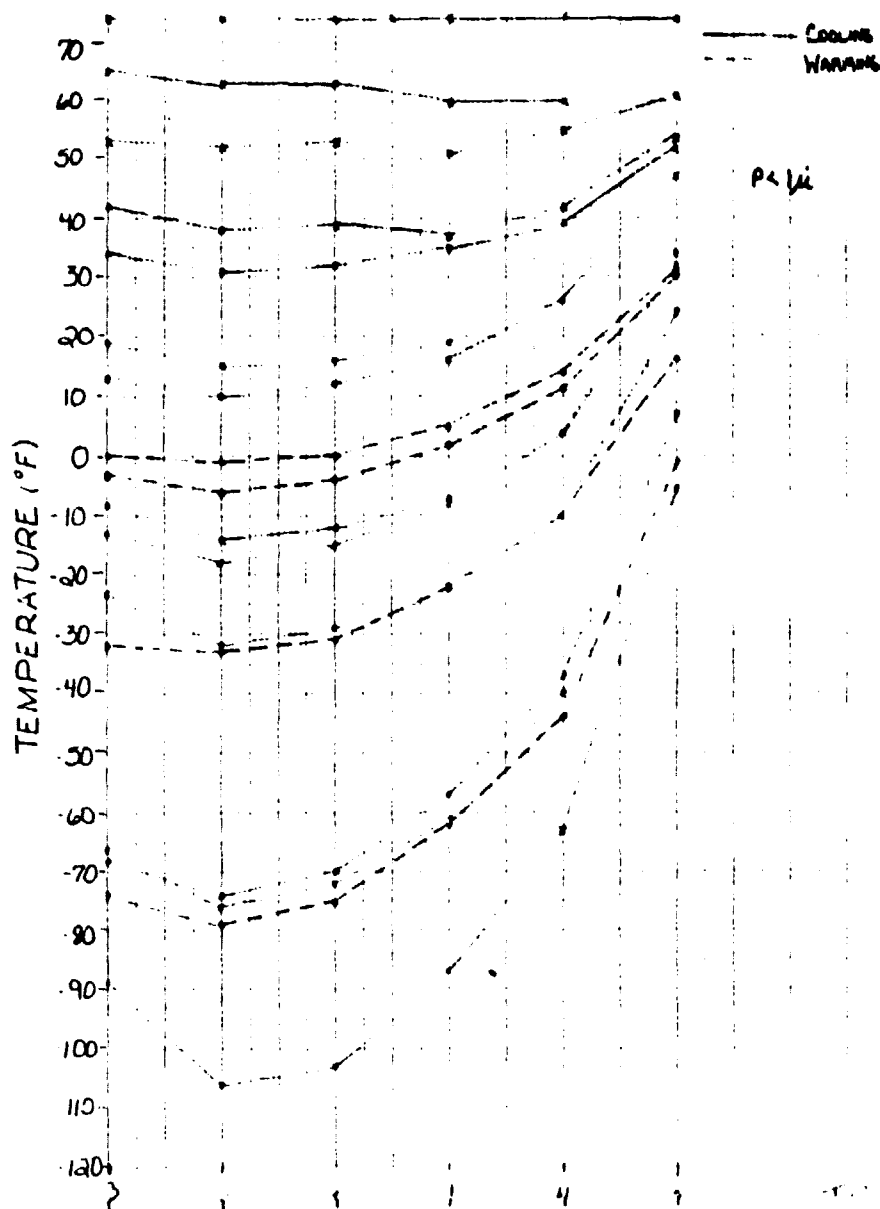


Figure 15 and 16 - Thermal profile along the back (i.e. away from the laser) half of the Graphite Epoxy sample under increasing temperature.



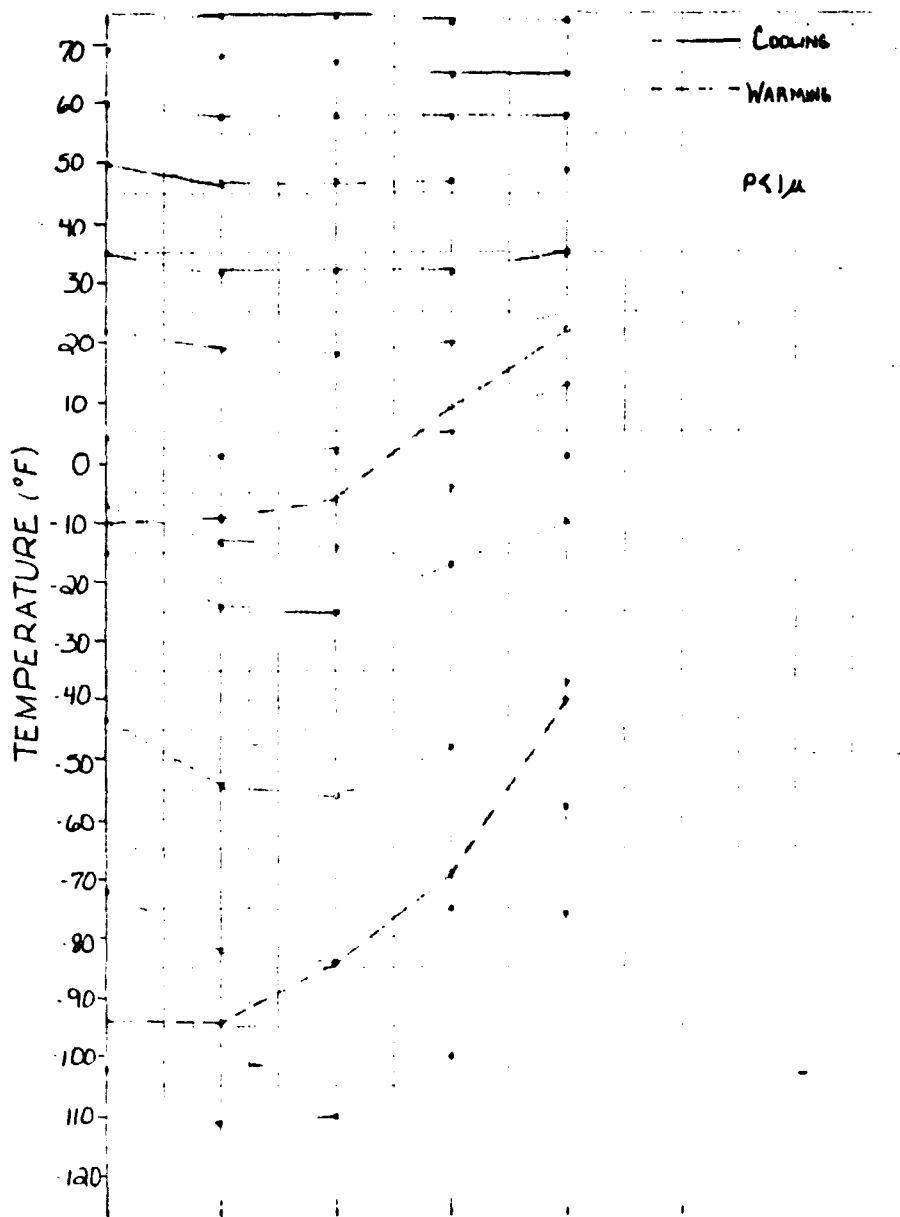
APPROXIMATE DISTANCE ALONG SAMPLE

Figure 17 - Thermal profile along the back (i.e. away from the laser) half of the Graphite Epoxy sample under the usual vacuum ( $< 1\mu H_g$ ).



APPROXIMATE DISTANCE ALONG SAMPLE

Figure 18 - Thermal profile along the front (i.e. the laser end) half of the Graphite Epoxy sample under the usual vacuum ( $< 1 \mu H_g$ ).



APPROXIMATE DISTANCE ALONG SAMPLE

Figure 19 - Thermal profile along the front (i.e. the laser end) half of the Graphite Epoxy sample under the usual vacuum ( $< 1\mu H_g$ ).



### VENDORS

Theta Industries, Inc. offers an eighty thousand dollar laser thermal expansion system using a similar H.P. laser. Some off-the-shelf components of that system may be very valuable to this M&P Lab/MSFC H.P. laser measuring system which is limited to near ambient by retroreflector mounts.

### CRITIQUE

If the measurements were repeated the following adjustments would be advisable.

1. Use smaller T.C. wire (< 5 mill)
2. Use 4 inlets (with 4 variable valves) to form 4 independent heating and cooling coils for each quarter of the sample.
3. Anneal the quartz at rod "B" at 1145°C then recheck  $\alpha$ .
4. Check CTE with retroreflectors only 1 cm apart.
5. Apply  $\sim 50$  thermal cycles to the G/E in a one or two day period.

## CONCLUDING THOUGHTS

We are not too happy with quartz as an absolute CTE standard. The larger than expected quartz CTE values could be due to relatively large thermocouples having 40 x 60 mil insulation over 12.5 mil wire, forcing the junction slightly toward ambient. Also with these thermocouples mounted on the bottom of the sample some bowing could also contribute to the increased CTE.

The ambient (20°C) CTE of quartz taken from the Amersil specification sheet is  $2.4 \times 10^{-7}/^{\circ}\text{F}$ . The  $3 \times 10^{-7}/^{\circ}\text{F}$  used here is the usually quoted value for quartz, but evidently refers to slightly higher temperatures. Thus the error in Figures 6, 6a, and 6b is about 30% larger than shown.

We feel 90% confident that our G/E CTE results do not error by more than -20% or +50% from 0 to 125°F. Below 0°F this confidence drops to 80% for the following reason. With the CTE decreasing with temperature for most materials, including quartz and G/E, the true CTE at the center temperature is slightly smaller than the stated value due to warmer parts of the sample having an exaggerated contribution, e.g. see Figures 12 through 18. If the outside 30% of the quartz sample has a 1.5 x larger CTE due to a 60°F higher temperature, then our measured CTE would be 1.15 x too high in some temperature range. If the outside 20% of the G/E sample has a 5 x larger CTE due to a 60°F higher temperature there, then (around -25°F) our measured CTE would be about 2 x too large. Below -40°F the ideal strain vs. T curves should flatten very much, since ours does not flatten, the CTE is not approaching zero as fast as considered above and the 2 x may be 1.4 x or less.

The ideal  $\Delta L$  vs.  $T$  curve for G/E has a knee around  $0^{\circ}\text{F}$  just below this knee  $-25 \pm 25^{\circ}\text{F}$  is where the above 1.4 x contribution should be greatest. At other temperatures the contribution is probably negligible as in the case of quartz. In fact the CTE is slightly smaller starting at near ambient. In other words some of the  $\Delta L$  decrease which should have occurred near ambient is occurring where the center temperature is about  $0^{\circ}\text{F}$ . This means that each run should extend down to  $-100^{\circ}\text{F}$  to see if a knee is there and only then could a correction be applied at about  $-25^{\circ}\text{F}$ .

The ambient overnight recovery to the original sample length shown in Fig. 7 C is comforting from the standpoint of building long life space structures. That is the length will probably not tend to "walk" or creep indefinitely for more than one or two PPM, in spite of the fact that the cycle is over 20 to 30 PPM, if allowed to recover at ambient for about a day. The above reversibility (on a 20 hour time scale) contradicts the irreversibility findings of Eselun et. al.<sup>6</sup> for one term of the equation on page 30. However, they may not be measuring on a 24 hour time scale and their sample is slightly different from ours.

#### CONCLUSION

Through this report doubts about the overall CTE system have been raised for consideration, however all in all the changing CTE measurements are thought to be rather accurate (+50% to -20%) and the differences from run to run, and in comparison to the vendor, are thought due to G/E sample moisture content variation. The sample had been removed from its wrapping (paper) about 3 months prior to run 1 in Fig. 4.

# REFERENCES

1. Hahn, T. A and Kirby, R. K., A.I.P. Conf. Proceedings, No. 3  
Thermal Expansion - 1971; Gram, M. G. and Hagy, H. E., Eds.,  
Amer. Inst. of Phy., N. Y. (1972) p. 13.
2. Wolff, E. M., "Materials and Processing - In Service Performance,"  
National SAMPE Tech. Conf. Series, V.9, pp 57-72 (1977).
3. Freund, N. P., Composite Reliability, ATSM, STP 580, American  
Society for Testing Materials, 1975, p. 133-145.
4. Wolff, E. M., and Eseluni, S. A., Review of Scientificate  
Instruments, p. 501, V. 50, No. 4, AP '79.
5. Bruckner, R., J. Non-Crystalline Solids, 5, 123-176, 177-216, 1970.
6. Eselun, S. A., Neubert, H. D., and Wolff, E. G., SAMPE, May  
1979.